



UNITED STATES PATENT AND TRADEMARK OFFICE

*In Re* Appl. of: Robert J. SMALL and Zhefei J. CHEN. Confirmation No: 1702  
Serial No: 10/057,206 Group: 1755  
Filed: January 25, 2002 Examiner: M. Marcheschi  
Entitled: **COMPOSITIONS FOR CHEMICAL-MECHANICAL PLANARIZATION OF NOBLE-METAL-FEATURED SUBSTRATES, ASSOCIATED METHODS, AND SUBSTRATES PRODUCED BY SUCH METHODS** ATTY. DOCKET: 63254-5002-US

**DECLARATION OF ROBERT J. SMALL AND ZHEFEI J. CHEN**  
**UNDER 37 C.F.R. § 1.131**

We, ROBERT J. SMALL, and ZHEFEI J. CHEN declare under the penalty of perjury as set forth below:

1. We are co-inventors named in the above-referenced patent application 10/057,206 which was filed on January 25, 2002 ("Application").
2. We were employed by EKC Technologies Inc., (hereinafter "EKC") the original Assignee ("Assignee") of the Application by virtue of an assignment recorded in the U.S. Patent Office at Reel 12540 and Frame 0635. As part of the work for EKC, we developed compositions for polishing a substrate having a noble metal material, or a material comprising a noble metal, on its surface. The compositions comprised periodic acid, or  $H_5IO_6$ , which was often referred to as "PIA" and which is referred to hereinafter as PIA, and an abrasive. Portions of this work are the subject matter disclosed in the Application and are the subject matter of claims 77-143 that are pending in this Application.
3. Our work for EKC was performed in the United States of America.
4. Pending claims 77-139 stand rejected in view of US Patent Application No. 2003/0153184 (Wang), which claims priority by way of a divisional application No. 09/636,246 filed on August 10, 2000, which in turn claims priority to a provisional application No. 60/148,813 filed on August 13, 1999, either alone or in view of U.S. Patent 6,783,434 (Akahori et al.), published application US 20020042208 ("Beitel"), published application US 20010044264 ("Lack"), and published application US 20020111027 ("Sachan").

**A: REDUCTION TO PRACTICE OF PENDING CLAIMS 77-143 BEFORE AUGUST 13, 1999**

5. As part of our work for EKC, we developed compositions for polishing a substrate having a noble metal material, or a material comprising a noble metal, on its surface. The compositions comprised periodic acid, or  $H_5IO_6$ , which we often referred to as "PIA" and which is referred to hereinafter as PIA, and an abrasive.

6. In certain embodiments, these components were present in the compositions in a combined amount to render the noble metal substrate surface substantially planar upon chemical-mechanical polishing of the substrate surface.

7. In certain embodiments, the periodic acid was present in the compositions in an amount from about 0.05 to about 0.3 moles/kg.

8. In certain embodiments, the composition had a pH of from about pH 1 to less than pH 2 or from above pH 5 to about pH 10.

9. In certain embodiments, the abrasive was present in the compositions in an amount of from about 0.2 to about 6 weight percent or from about 0.2 to about 4 weight percent.

10. We believe that the subject matter set forth above in items 5-9, as well as other subject matter set forth herein, was *conceived and reduced to practice before August 13, 1999 (the effective filing date of the Wang reference)*.

11. Pending claims 77-93 of the instant Application relate to the subject matter set forth above in items 5-9, the reduction to practice of which is described below in relation to claim 77.

12. Pending claims 94-111 of the Application relate to the subject matter set forth above in items 5-9, the reduction to practice of which is described below in relation to claim 94.

13. Pending claims 118-143 of the Application relate to the subject matter set forth

above in items 5-9, the reduction to practice of which is described below in relation to claim 118.

14. Appended hereto in Appendix 1 are (date redacted) copies of two pages from a Cleanroom notebook of Zhefei J. Chen concerning the chemical-mechanical polishing of two wafers, one wafer having a noble metal material, iridium (Ir), and the other wafer having a noble metal oxide material, iridium oxide (IrO<sub>2</sub>), on its surface.

15. The chemical-mechanical polishing (CMP) was carried out using a composition comprising about 2 weight percent of a pure  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> abrasive, which we often referred to as "CRI" and is referred to herein as CRI, and (0.1 mole of PIA in a 300g composition), and having a pH of about 0.5 to 1. Upon chemical mechanical polishing of the wafers, the latter wafer was shown to be edge polished, shining, and uniform, which was considered a positive result. The content of the two pages was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

16. Appended hereto in Appendix 2 is a (date redacted) copy of a page of a Cleanroom notebook of Zhefei J. Chen concerning a chemical mechanical polishing composition for polishing wafers, each having a material comprising a noble metal material on its surface. The composition, which we often referred to as "ZCX200" and is referred to herein as ZCX200, comprised about 2 weight percent of an alumina abrasive, the commercial product "CR-30" manufactured by Baikowski Chimie Co. of Annecy Cedex 9, France, which is referred to herein as CR30, and about 0.1 mole/kilogram of PIA. ZCX200 was prepared to have a pH of about 1.5, or a pH of about 6 to about 7.5, measured either by probe or meter, or by paper, usually brought about by the addition of tetramethylammonium hydroxide (TMAH) and/or aminonium hydroxide (NH<sub>4</sub>OH). Also attached to this page is another page showing CMP compositions with 4% CR30 (alumina) and 0.1 moles/kg of periodic acid. The content of these pages were prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

17. Appended hereto in Appendix 3 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen concerning the chemical-mechanical polishing of a number of wafers, each having iridium (Ir) on its surface. For three of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5) (the reference to a pH of  $\leq 2.5$  was used to indicate the expected pH, while the parenthetic reference

to a pH of 1.5 was used to indicate the actual pH, as measured), and for another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+). The pH of the solution was adjusted using either ammonium hydroxide (NH<sub>4</sub>OH) or tetra-methyl-ammonium-hydroxide (TMAH). The chemical-mechanical polishing of each of the wafers was associated with a removal rate of 288, 400, 375, and 324 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

18. The page mentioned above in item 17 also concerned the chemical-mechanical polishing of three wafers, each having Iridium (Ir) on its surface. The chemical-mechanical polishing was carried out using a composition comprising about 2 weight percent of CR30 abrasive and about 0.1 to 0.2 mole/kilogram of PIA, and having a pH of about 1.5 (the reference to a pH of ~5 was used to indicate the expected pH, while the parenthetical reference to a pH of 1.5 was used to indicate the actual pH, as measured). The chemical-mechanical polishing of each of the wafers was associated with a removal rate of 250, 275, and 350 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

19. The page mentioned above in item 17 also concerned the chemical-mechanical polishing of another wafer having Ir on its surface. The chemical-mechanical polishing was carried out using a composition comprising about 4 weight percent of CR30 abrasive and about 0.1 mole/kilogram of PIA, and having a pH of about 1.5 (the reference to a pH of ≤ 2.5 was used to indicate the expected pH, while the parenthetical reference to a pH of 1.5 was used to indicate the actual pH, as measured). The chemical mechanical polishing of the wafer was associated with a removal rate of 290 Å/min, which was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

20. Appended hereto in Appendix 4 is a (date redacted) copy of a page of a cleanroom notebook of Zhefei J. Chen concerning the chemical mechanical polishing of a number of wafers, each having Ir on its surface. For four of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+), for which the pH was 7, as measured by meter, and 7.75, as measured by paper. The chemical-mechanical polishing of each of the wafers was associated with a roughness data (RMS data) of 7.28, 6.68, 5.64, and 6.47 Å, respectively, each

of which was considered a positive result. The content of the page was prepared before *before August 13, 1999 (the effective filing date of the Wang reference)*.

21. Appended hereto in Appendix 5 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. For one of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+); for another of the wafers, the chemical-mechanical polishing was carried out using a composition comprising 2 weight percent of CR30 abrasive and about 0.2 mole/kilogram of PIA, and having a pH of about 7; and for yet another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5). The chemical mechanical polishing of each of the wafers was associated with roughness data (RMS or Scratch data) as set forth in the appended page, respectively, each of which was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

22. Appended hereto in Appendix 6 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a Ir and IrO<sub>2</sub>. For these wafers, the CMP was carried out using CR30 abrasive with 2% by weight, and 0.1 moles/kg of PIA at a pH of 6-7 adjusted with TMAH. For IrO<sub>2</sub>, the CMP was carried out with CR30, 2% by weight of abrasive, and hydrazine with 0.05 moles/kg at a pH of 9. The CMP was carried out at various applied pressure ranging from 2 to 6 psi. The polishing results (removal rate) range from 100 to 450 Å/min for the Ir and 355 to 1116 Å for IrO<sub>2</sub>. The chemical mechanical polishing results as set forth in the graphs, respectively, was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

23. Appended hereto in Appendix 7 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the CMP of Iridium on TEOS (dielectric) and its polishing results using periodic acid and various pH ranging from 7 to 12. The CMP was carried out with 2 wt% of abrasive and 0.1 moles/kg of periodic acid at a pH ranging from 9-12. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

24. Appended hereto in Appendix 8 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the CMP of Iridium and its polishing results using 2 wt% of abrasive and periodic acid (0.1 mol/kg) at various pH ranging from 7 to 12. This table illustrates the use of organic acid (oxalic acid) including pentadione in the CMP slurry. Appended here to is another (date redacted) page showing that oxalic acid (a suspension agent) is added to the CMP composition to enhance complexing. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

25. Appended hereto in Appendix 9 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the use of various abrasives for the test results. The list includes  $\text{Al}_2\text{O}_3$ , MgO including a mixture of cerium and alumina. The appended page to this exhibit shows the use of silica as another abrasive. The content of these pages was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

26. Appended hereto in Appendix 10 is a (date redacted) copy of pages that were appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the conception of polishing platinum using periodic acid. The content of these pages was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

27. The above-mentioned pages were generated from chemical-mechanical polishing experiments implemented by Zhefei J. Chen with the assistance of Bob Small's direct supervision *before August 13, 1999 (the effective filing date of the Wang reference)*.

28. The above-mentioned pages and similar information dated **both before and after August 13, 1999** have been kept among the usual business records of the Assignee in the ordinary course of business.

29. In view of the foregoing, we believe the invention as reflected in independent claims 77, 94, and 118 including claims depending therefrom of this instant Application was conceived and reduced to practice *before August 13, 1999 (the effective filing date of the Wang reference)*.

**B: DILIGENCE PERIOD FROM AUGUST 1999 to JANUARY 25, 2002 (FILING DATE)**

Applicants worked diligently from the date prior to *August 13, 1999 (the effective filing date of the Wang reference)* until the actual filing date of this application on January 25, 2002 to do the following: a) further refine and conduct CMP research with other noble metals; b) review and analyze the complex data and results internally on this subject research project on CMP of noble metal polishing using periodic acid including with other third-party scientists; 3) find and hire an outside patent agent to prepare draft disclosures on several CMP projects completed at EKC; 4) contact an outside law firm to prepare, revise, and finalize the draft applications and file the application on January 25, 2002. Below is Applicants timeline of the events and circumstance showing Applicants' "**reasonable diligence**" since the reduction to practice to the actual filing date of the invention on January 25, 2002.

30. After finishing Iridium and IrO<sub>2</sub> polishing using periodic acid, our research efforts were being focused on polishing other noble metals such as platinum (Pt) including copper (Cu) using CMP compositions comprising similar or different abrasives and periodic acid until the end of December 1999. Attached hereto is an **Exhibit 11**, showing a cleanroom lab notebook page of Zhefei Chen from September 28, 1999 on polishing Platinum using periodic acid in combination with TMAH and ammonium hydroxide. Attached hereto is an **Exhibit 12** showing a cleanroom lab notebook page of Zhefei Chen from **October 1, 1999**, on polishing copper wafers. Attached hereto is an **Exhibit 13** showing a cleanroom lab notebook page of Zhefei Chen from **November 24, 1999** on polishing Platinum on BPSG (borophosphosilicate) using periodic acid. Attached thereto is another cleanroom lab notebook page of Zhefei Chen from **December 15, 1999** on polishing results of platinum using periodic acid and a suspending agent (Alumina-C). The chemical-mechanical polishing composition comprised about 6 weight percent of CR30 abrasive and a commercial alumina abrasive "Alumina-C" manufactured by Deguss-Huls AG, which is referred to herein as Alumina-C, and about 0.1 mole/kilogram of PIA, which had a pH of about 1.75. The chemical-mechanical polishing composition was kept overnight and evaluated as to its suspension characteristics, which were considered positive.

31. From **January 2000 through April 2000**, as part of our business practice, our senior managers at EKC reviewed our research results on noble metal polishing using periodic

acid internally with other scientists at our site at EKC, Hayward, CA. During this time, we also discussed our work of noble metal polishing using periodic acid with third party scientists. Realizing that periodic acid worked very well for CMP polishing of noble metals, our senior managers decided to pursue bringing our research work into the public domain.

32. Around **March 2000**, our senior managers at EKC decided to push the momentum and accelerate preparing and filing several patent disclosures of EKC's research work on all CMP projects conducted before 2000. There were at least six CMP research projects that were mostly complete by the end of 1999 for which EKC decided to pursue filing patent applications. The research project on CMP of noble metal polishing using periodic acid (the instant invention) was one of the six research projects that our senior managers identified and decided to pursue filing a patent application. Among the six inventions, this instant invention for polishing noble metals was placed in order of priority with other CMP inventions on polishing copper, tantalum, and tungsten, which were conducted and completed at EKC at an earlier time.

33. On or about **April 2000**, Bob Small, Research Director at EKC, contacted an outside patent agent, Dennis Deboo, an independent and solo patent agent, who had an electrical engineering background to prepare patent disclosures for EKC with the intent to get all the completed CMP projects at EKC into the public domain.

34. From about **April 2000** to about **March 2001**, Dennis Deboo met with Bob Small on a number of occasions to discuss the six patent disclosures from EKC including the disclosure of this instant invention. Mr. Deboo started preparing draft disclosures in **April of 2000**. He completed the first disclosure on "Compositions for Cleaning CMP Apparatus" on May 18, 2000. It was customary practice for Mr. Deboo to prepare a draft a disclosure one at a time. Once he prepared a draft disclosure, he would customarily send the completed draft disclosures to Dr. Small for review and then to the law firm of Skervin, Morrill, Macpherson, Franklin & Friel for further revisions and filing. We believe that the instant application was in "queue" or in "sequence" at Mr. Deboo's drafting docket along with five other applications from EKC. Attached as **Exhibit 14** is Mr. Deboo's docket sheet showing the "queue" of patent applications from EKC. The instant application was number 6 in this docket. We believe that given the fact that Mr. Deboo was a solo practitioner who was also engaged in drafting other patent disclosures from other clients concurrently, Ms. Deboo was very diligent in preparing the EKC disclosures.



35. We believe that Mr. Deboo started to prepare a draft disclosure on this instant invention on or about **May 2001** after having finished five other applications for EKC that were in queue, in chronological order, before this application. His turn-around time for this application was about six to eight weeks. His records indicate that he completed the draft disclosure on **July 24, 2001**. Attached as **Exhibit 15** is a (redacted) letter from Mr. Deboo showing that he completed preparing a draft application on July 24, 2001. Also attached to this exhibit are sample pages of the draft application completed on **July 24, 2001**.

36. After Mr. Deboo received instructions from Dr. Small, Mr. Deboo sent a draft application (also in a disk) to the law firm of Skerven, Morrill, Macpherson, Franklin & Friel for further revision and filing on **August 22, 2001**. See, **Exhibit 16**, a redacted copy of the letter from Mr. Deboo addressed to George Wolken Jr. at Skerven, Morrill, Macpherson, Franklin & Friel including a draft application having a total of 47 numbered pages titled, "COMPOSITIONS FOR NOBEL METAL CHEMICAL-MECHANICAL PLANARIZATION PROCESSES draft application dated August 22, 2001.

37. On **October 1, 2001** a first draft application was prepared by the law firm of Skerven, Morrill, Macpherson, Franklin & Friel. Attached as **Exhibit 17** is a redacted communication from George Wolken to Dr. Small indicating the completion and transmittal of the draft application for review for Dr. Small. Also attached is a copy of the first draft application prepared by this firm.

38. On **November 16, 2001** a second draft application was prepared by patent agent Ms. Alison De Runtz and forwarded to Dr. Small. Because of the complex subject matter of this invention Dr. Small and Ms. De Runtz engaged in frequent communications regarding the technical aspects and claims of this invention. Attached as **Exhibit 18** is a redacted copy of an e-mail from Ms. De Runtz to Bob Small regarding transmittal of this second draft application, including a communication dated **November 26, 2001** regarding preparing a "third" draft application.

39. On **December 20, 2001** another revised draft application (third) was prepared by Ms. De Runtz and sent to Dr. Small for review. Attached as **Exhibit 19** is a redacted copy of the communication from Ms. De Runtz to Dr. Small on this revised draft application based on the new material supplied by Dr. Small.

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40. On January 18, 2002 a "ready to file" patent application was prepared and sent to Dr. Small by Alison De Runtz. A redacted copy of that letter sent to Dr. Small is attached here as Exhibit 20.

41. On January 24, 2002, the instant application was filed at the USPTO.

42. In view of the foregoing evidence on conception and reduction to practice including reasonable diligence, the invention as reflected in the pending claims 77-143 was conceived and reduced to practice before August 13, 1999 (the effective filing date of the Wang reference).

43. We further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: 5/23/08

  
Robert J. Small

Dated: \_\_\_\_\_

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Zheifei J Chen



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**DECLARATION OF ROBERT J. SMALL AND ZHEFEI J. CHEN**  
**UNDER 37 C.F.R. § 1.131**

We, ROBERT J. SMALL, and ZHEFEI J. CHEN declare under the penalty of perjury as set forth below:

1. We are co-inventors named in the above-referenced patent application 10/057,206 which was filed on January 25, 2002 ("Application").
2. We were employed by EKC Technologies Inc., (hereinafter "EKC") the original Assignee ("Assignee") of the Application by virtue of an assignment recorded in the U.S. Patent Office at Reel 12540 and Frame 0635. As part of the work for EKC, we developed compositions for polishing a substrate having a noble metal material, or a material comprising a noble metal, on its surface. The compositions comprised periodic acid, or  $\text{H}_5\text{IO}_6$ , which was often referred to as "PIA" and which is referred to hereinafter as PIA, and an abrasive. Portions of this work are the subject matter disclosed in the Application and are the subject matter of claims 77-143 that are pending in this Application.
3. Our work for EKC was performed in the United States of America.
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**A: REDUCTION TO PRACTICE OF PENDING CLAIMS 77-143 BEFORE AUGUST 13, 1999**

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6. In certain embodiments, these components were present in the compositions in a combined amount to render the noble metal substrate surface substantially planar upon chemical-mechanical polishing of the substrate surface.

7. In certain embodiments, the periodic acid was present in the compositions in an amount from about 0.05 to about 0.3 moles/kg.

8. In certain embodiments, the composition had a pH of from about pH 1 to less than pH 2 or from above pH 5 to about pH 10.

9. In certain embodiments, the abrasive was present in the compositions in an amount of from about 0.2 to about 6 weight percent or from about 0.2 to about 4 weight percent.

10. We believe that the subject matter set forth above in items 5-9, as well as other subject matter set forth herein, was *conceived and reduced to practice before August 13, 1999 (the effective filing date of the Wang reference)*.

11. Pending claims 77-93 of the instant Application relate to the subject matter set forth above in items 5-9, the reduction to practice of which is described below in relation to claim 77.

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14. Appended hereto in **Appendix 1** are (date redacted) copies of two pages from a Cleanroom notebook of Zhefei J. Chen concerning the chemical-mechanical polishing of two wafers, one wafer having a noble metal material, iridium (Ir), and the other wafer having a noble metal oxide material, iridium oxide (IrO<sub>2</sub>), on its surface.

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of which was considered a positive result. The content of the page was prepared before *before August 13, 1999 (the effective filing date of the Wang reference)*.

21. Appended hereto in Appendix 5 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. For one of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+); for another of the wafers, the chemical-mechanical polishing was carried out using a composition comprising 2 weight percent of CR30 abrasive and about 0.2 mole/kilogram of PIA, and having a pH of about 7; and for yet another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5). The chemical mechanical polishing of each of the wafers was associated with roughness data (RMS or Scratch data) as set forth in the appended page, respectively, each of which was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

22. Appended hereto in Appendix 6 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a Ir and IrO<sub>2</sub>. For these wafers, the CMP was carried out using CR30 abrasive with 2% by weight, and 0.1 moles/kg of PIA at a pH of 6-7 adjusted with TMAH. For IrO<sub>2</sub>, the CMP was carried out with CR30, 2% by weight of abrasive, and hydrazine with 0.05 moles/kg at a pH of 9. The CMP was carried out at various applied pressure ranging from 2 to 6 psi. The polishing results (removal rate) range from 100 to 450 Å/min for the Ir and 355 to 1116 Å for IrO<sub>2</sub>. The chemical mechanical polishing results as set forth in the graphs, respectively, was considered a positive result. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

23. Appended hereto in Appendix 7 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the CMP of Iridium on TEOS (dielectric) and its polishing results using periodic acid and various pH ranging from 7 to 12. The CMP was carried out with 2 wt% of abrasive and 0.1 moles/kg of periodic acid at a pH ranging from 9-12. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

24. Appended hereto in Appendix 8 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the CMP of Iridium and its polishing results using 2 wt% of abrasive and periodic acid (0.1 mol/kg) at various pH ranging from 7 to 12. This table illustrates the use of organic acid (oxalic acid) including pentadione in the CMP slurry. Appended here to is another (date redacted) page showing that oxalic acid (a suspension agent) is added to the CMP composition to enhance complexing. The content of the page was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

25. Appended hereto in Appendix 9 is a (date redacted) copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the use of various abrasives for the test results. The list includes Al<sub>2</sub>O<sub>3</sub>, MgO including a mixture of cerium and alumina. The appended page to this exhibit shows the use of silica as another abrasive. The content of these pages was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

26. Appended hereto in Appendix 10 is a (date redacted) copy of pages that were appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the conception of polishing platinum using periodic acid. The content of these pages was prepared *before August 13, 1999 (the effective filing date of the Wang reference)*.

27. The above-mentioned pages were generated from chemical-mechanical polishing experiments implemented by Zhefei J. Chen with the assistance of Bob Small's direct supervision *before August 13, 1999 (the effective filing date of the Wang reference)*.

28. The above-mentioned pages and similar information dated **both before and after August 13, 1999** have been kept among the usual business records of the Assignee in the ordinary course of business.

29. In view of the foregoing, we believe the invention as reflected in independent claims 77, 94, and 118 including claims depending therefrom of this instant Application was conceived and reduced to practice *before August 13, 1999 (the effective filing date of the Wang reference)*.



**B: DILIGENCE PERIOD FROM AUGUST 1999 to JANUARY 25, 2002 (FILING DATE)**

Applicants worked diligently from the date prior to *August 13, 1999 (the effective filing date of the Wang reference)* until the actual filing date of this application on January 25, 2002 to do the following: a) further refine and conduct CMP research with other noble metals; b) review and analyze the complex data and results internally on this subject research project on CMP of noble metal polishing using periodic acid including with other third-party scientists; 3) find and hire an outside patent agent to prepare draft disclosures on several CMP projects completed at EKC; 4) contact an outside law firm to prepare, revise, and finalize the draft applications and file the application on January 25, 2002. Below is Applicants timeline of the events and circumstance showing Applicants' "**reasonable diligence**" since the reduction to practice to the actual filing date of the invention on January 25, 2002.

30. After finishing Iridium and IrO<sub>2</sub> polishing using periodic acid, our research efforts were being focused on polishing other noble metals such as platinum (Pt) including copper (Cu) using CMP compositions comprising similar or different abrasives and periodic acid until the end of December 1999. Attached hereto is an **Exhibit 11**, showing a cleanroom lab notebook page of Zhefei Chen from September 28, 1999 on polishing Platinum using periodic acid in combination with TMAH and ammonium hydroxide. Attached hereto is an **Exhibit 12** showing a cleanroom lab notebook page of Zhefei Chen from **October 1, 1999**, on polishing copper wafers. Attached hereto is an **Exhibit 13** showing a cleanroom lab notebook page of Zhefei Chen from **November 24, 1999** on polishing Platinum on BPSG (borophosphosilicate) using periodic acid. Attached thereto is another cleanroom lab notebook page of Zhefei Chen from **December 15, 1999** on polishing results of platinum using periodic acid and a suspending agent (Alumina-C). The chemical-mechanical polishing composition comprised about 6 weight percent of CR30 abrasive and a commercial alumina abrasive "Alumina-C" manufactured by Deguss-Huls AG, which is referred to herein as Alumina-C, and about 0.1 mole/kilogram of PIA, which had a pH of about 1.75. The chemical-mechanical polishing composition was kept overnight and evaluated as to its suspension characteristics, which were considered positive.

31. From **January 2000 through April 2000**, as part of our business practice, our senior managers at EKC reviewed our research results on noble metal polishing using periodic

acid internally with other scientists at our site at EKC, Hayward, CA. During this time, we also discussed our work of noble metal polishing using periodic acid with third party scientists. Realizing that periodic acid worked very well for CMP polishing of noble metals, our senior managers decided to pursue bringing our research work into the public domain.

32. Around **March 2000**, our senior managers at EKC decided to push the momentum and accelerate preparing and filing several patent disclosures of EKC's research work on all CMP projects conducted before 2000. There were at least six CMP research projects that were mostly complete by the end of 1999 for which EKC decided to pursue filing patent applications. The research project on CMP of noble metal polishing using periodic acid (the instant invention) was one of the six research projects that our senior managers identified and decided to pursue filing a patent application. Among the six inventions, this instant invention for polishing noble metals was placed in order of priority with other CMP inventions on polishing copper, tantalum, and tungsten, which were conducted and completed at EKC at an earlier time.

33. On or about **April 2000**, Bob Small, Research Director at EKC, contacted an outside patent agent, Dennis Deboo, an independent and solo patent agent, who had an electrical engineering background to prepare patent disclosures for EKC with the intent to get all the completed CMP projects at EKC into the public domain.

34. From about **April 2000** to about **March 2001**, Dennis Deboo met with Bob Small on a number of occasions to discuss the six patent disclosures from EKC including the disclosure of this instant invention. Mr. Deboo started preparing draft disclosures in **April of 2000**. He completed the first disclosure on "Compositions for Cleaning CMP Apparatus" on May 18, 2000. It was customary practice for Mr. Deboo to prepare a draft a disclosure one at a time. Once he prepared a draft disclosure, he would customarily send the completed draft disclosures to Dr. Small for review and then to the law firm of Skervin, Morrill, Macpherson, Franklin & Friel for further revisions and filing. We believe that the instant application was in "queue" or in "sequence" at Mr. Deboo's drafting docket along with five other applications from EKC. Attached as **Exhibit 14** is Mr. Deboo's docket sheet showing the "queue" of patent applications from EKC. The instant application was number 6 in this docket. We believe that given the fact that Mr. Deboo was a solo practitioner who was also engaged in drafting other patent disclosures from other clients concurrently, Ms. Deboo was very diligent in preparing the EKC disclosures.

35. We believe that Mr. Deboo started to prepare a draft disclosure on this instant invention on or about **May 2001** after having finished five other applications for EKC that were in queue, in chronological order, before this application. His turn-around time for this application was about six to eight weeks. His records indicate that he completed the draft disclosure on **July 24, 2001**. Attached as **Exhibit 15** is a (redacted) letter from Mr. Deboo showing that he completed preparing a draft application on July 24, 2001. Also attached to this exhibit are sample pages of the draft application completed on **July 24, 2001**.

36. After Mr. Deboo received instructions from Dr. Small, Mr. Deboo sent a draft application (also in a disk) to the law firm of Skerven, Morrill, Macpherson, Franklin & Friel for further revision and filing on **August 22, 2001**. See, **Exhibit 16**, a redacted copy of the letter from Mr. Deboo addressed to George Wolken Jr. at Skerven, Morrill, Macpherson, Franklin & Friel including a draft application having a total of 47 numbered pages titled, "COMPOSITIONS FOR NOBEL METAL CHEMICAL-MECHANICAL PLANARIZATION PROCESSES draft application dated August 22, 2001.

37. On **October 1, 2001** a first draft application was prepared by the law firm of Skerven, Morrill, Macpherson, Franklin & Friel. Attached as **Exhibit 17** is a redacted communication from George Wolken to Dr. Small indicating the completion and transmittal of the draft application for review for Dr. Small. Also attached is a copy of the first draft application prepared by this firm.

38. On **November 16, 2001** a second draft application was prepared by patent agent Ms. Alison De Runtz and forwarded to Dr. Small. Because of the complex subject matter of this invention Dr. Small and Ms. De Runtz engaged in frequent communications regarding the technical aspects and claims of this invention. Attached as **Exhibit 18** is a redacted copy of an e-mail from Ms. De Runtz to Bob Small regarding transmittal of this second draft application, including a communication dated **November 26, 2001** regarding preparing a "third" draft application.

39. On **December 20, 2001** another revised draft application (third) was prepared by Ms. De Runtz and sent to Dr. Small for review. Attached as **Exhibit 19** is a redacted copy of the communication from Ms. De Runtz to Dr. Small on this revised draft application based on the new material supplied by Dr. Small.

40. On January 18, 2002 a "ready to file" patent application was prepared and sent to Dr. Small by Alison De Runtz. A redacted copy of that letter sent to Dr. Small is attached here as Exhibit 20.

41. On January 24, 2002, the instant application was filed at the USPTO.


42. In view of the foregoing evidence on conception and reduction to practice including reasonable diligence, the invention as reflected in the pending claims 77-143 was conceived and reduced to practice before *August 13, 1999 (the effective filing date of the Wang reference)*.

43. We further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated: \_\_\_\_\_

\_\_\_\_\_  
Robert J. Small

Dated: 5/27/08

  
\_\_\_\_\_  
Zheifei J Chen

EXHIBIT

tabbies

1

Notebook Number: \_\_\_\_\_

Project: \_\_\_\_\_

Irb-9-1  
 + CR1 + APS (-0.1 mole)  
 $APS = 0.1 \text{ mole} \times 228 \text{ g/mole} = 45.6 \text{ g}$  ~~77.7 g~~ since  $\frac{10}{3}$   
 CR1 = 6 g  
 DI = 202.4 g  
 pH = 0.5  
 1.06 563.18 11.60 Black on pad.

Ir02-19 1.06 12738.29 130.53 more black on pad  
 -19-1 remanence after 1 hr 10729.54 11.13

7/2/99 \* CR1 +  $H_5IO_7$  (periodic acid)  
 $PIA = 0.2 \text{ mole} \times 228 = 45.6 \text{ g}$  in 300 g 200 mmh.  
 Actually PIA used = 0.1 mole = 45.6 g in 300 g.  
 { PIA = 90 g from GFS Chemicals }  
 { CR1 = 12 g }  $\rightarrow$  pH = 0.5 - 1  
 DI = 498 g very soluble

Irb-9-1 recleaned  $\rightarrow$  562.10 11.33 12 mm  
 Ir02-19-2 recleaned  $\rightarrow$  11539.67 132.58 not broken yet

Ir031  $\rightarrow$  original npr.  $\rightarrow$  5773.68 2.98

next page

Read and Understood By:

Sig

Dat

Project: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

Initial measures for last page

Wfr I.D.	Slurry	Pressure	modulus	std. Dev.	Comments
Ir6-10	CR1+PIA PH=0.5-1	1.06	567.50	11.68	
Ir03-1	CR1+PIA	1.06	5783.28	3.09	Edge polished, shining. uniform

HAS from BASF  $(\text{NH}_4\text{OH})_2 \text{H}_2\text{SO}_4 = 2 \times 33 + 98 = 164$ HAS = 0.1 mole  $\times 164 = 16.4 \text{ g}$  for 300 g solids

Actual HAS : 32.8 g  
 CR1 : 12 g  
 DC : 555 g

pH 4 - 4.5  
 Dissolve easy

Ir03-2 CR1+HAS 1.06 5813.51 3.60 Edge polished more.

Ir6-11 CR1+HAS 1.06 56949 12.00

17/8/99

Ir7 original  $\longrightarrow$  515.42 3.17

Read and Understood By:

Project:

Notebook Number:

use TMAH

PIA  $\xrightarrow{\text{shift pH}}$  pH  $\xrightarrow{\text{use TMAH}}$  Caustic  $\sim 6$ ? W-case  
 (pH = 2.5) RR  $\uparrow$

H<sub>2</sub>O<sub>2</sub> - Ammonium, remove Pt

HDA - H<sub>2</sub>O<sub>2</sub> can not

	PIA ✓	CR30 ✓	DI ✓	TMAH	pH	probe	paper
① Tr	22.8g	20g	957g	<50ml	Initial 1.5	7.5	6.0
	H <sub>2</sub> O <sub>2</sub>	CR30 ✓	DI ✓	NH <sub>4</sub> OH	pH		
② Tr	10.9g	20g	969g	<20ml	Initial ~6	10.5	9.5
	PIA ✓	CR30 ✓	DI ✓				
③ Tr	45.6g	40g	1915g				

② H<sub>2</sub>O<sub>2</sub>

roject:

EXHIBIT

tabbles

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Create a new DOE on

Wfr type	Abrasive	Chemiscal	Psi	Platen rpm	Status
Ir	2% CR30	0.1 mol/Kg PIA	4	50	completed
	2% CR30	0.1 mol/Kg PIA	6	50	will do
	2% CR30	0.2 mol/Kg PIA	4	50	will do
	4% CR30	0.1 mol/Kg PIA	4	50	will do
	2% CR30	0.1 mol/Kg PIA	4	70?	will do
IrO2	2% CR30	0.1 mol/Kg NH2NH2	4	50	completed
	2% CR30	0.1 mol/Kg NH2NH2	6	50	will do
	2% CR30	0.2 mol/Kg NH2NH2	4	50	will do
	4% CR30	0.1 mol/Kg NH2NH2	4	50	will do
	2% CR30	0.1 mol/Kg NH2NH2	4	70?	will do

PIA + TMAH to pH6

H2O2 + amm to pH ?? (caustic side)

→ How about to pH ~ 10?

$H_2O_2 + CR6/CR30 \sim 2\% \rightarrow pH = 5-6$



EXHIBIT

3

Project: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

<u>Wfr. I.D.</u>	<u>Slurry</u>	<u>Psi/min</u>	<u>inches/SF</u>	<u>Std. Dev.</u>	<u>Comments</u>
Ir16	Unprocessed.	→	513.18	2.79%	
Ir16-1	CR30, H <sub>2</sub> O <sub>2</sub> + NH <sub>4</sub> OH PH = 9.5 by paper	4/5	545.79	2.60%	
Ir16-2	CR30, PIA + TMAH	4/5	3530	24.76%	several small cracks
Ir16-3	DI	6/5 / 50 rpm platen			No damage
Ir16-4	DI	4/70 rpm platen			No damage
Then safe to use higher rpm and pressure.					
Ir17	Unprocessed		514.01	2.81%	
Ir17-1	CR30, PIA	6/2 / 50 rpm platen	917.94	3.00%	very black on platen
Ir17-2	CR30, PIA	4/2 / 70 rpm platen	2456.2	12.57%	Very black on platen

New DOE on 7/27/99:

7/28/99 results

Wfr type	Abrasive	Chemical	pH	Psi	Platen rpm	RR (A/min)
Ir	2% CR30	0.1 mol/Kg PIA	≤ 2.5 (1.5)	4	50	288
	2% CR30	0.1 mol/Kg PIA	≤ 2.5 (1.5)	4	70	400
	2% CR30	0.1 mol/Kg PIA	≤ 2.5 (1.5)	6	50	375
	2% CR30	0.1 mol/Kg PIA + TMAH	6	4	50	324 ← start point, 1500/yr
	2% CR30	0.1 mol/Kg H <sub>2</sub> O <sub>2</sub> + NH <sub>4</sub> OH	9.5	4	50	10
	2% CR30	0.2 mol/Kg PIA <del>PIA + TMAH</del>	≤ 2.5	4	50	275, 250, 350
	4% CR30	0.1 mol/Kg PIA <del>PIA + TMAH</del>	≤ 2.5	4	50	290
IrO2	2% CR30	0.1 mol/Kg NH <sub>2</sub> NH <sub>2</sub>	9.0 - 9.5	4	50	880
	2% CR30	0.03 mol/Kg TMAH	9 - 10	4	50	> 320
	2% CR30	0.1 mol/Kg TMAH	10	4	50	> 635, 640
	2% CR30	0.05 mol/Kg NH <sub>2</sub> NH <sub>2</sub>	9	4	50	1740
	2% CR30	0.1 mol/Kg TMAH + 0.1 mol/Kg HDA/KS	11-12	4	50	> 420, 713

Read and Interpreted By: \_\_\_\_\_

Sigma

Date

Project: \_\_\_\_\_

EXHIBIT

tabbles

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Number: \_\_\_\_\_

I.D.	Spec	PH	m Oiling/eq.	Stk. Dev.	PSI/Rev/min	RMS
Ir 20	Unpaved		→ 511.62	2.80%		2.71A°
Ir 20-1	ZCX200	17	775 607.80 <i>note: paper</i>	2.51%	2/50/2min	7.25 2.81A°
Ir 20-2	"	"	825.23	4.41%	6/50/1min	7.28 A°
Ir 20-3	"	"	1075.5	7.40%	4/70/1min	5.64 A°
Ir 20-4	"	"	1515.1	13.62%	4/90/1min	6.47 A°
Ir 20-5	(CR30 + H400) 3% oil/mile		2245.9	22.21%	4/50/5min	scratches small
Ir 20	Unpaved		→ 515.31	4.21%		
Ir 21-1	ZCX200	7	712.03	2.58%	6/90/1min	7.45 A°, one bky

Signature

please sign on page!

Signature

Date

Date



Project:

Notebook Number:

8/9/99 Summary of Roughness data:

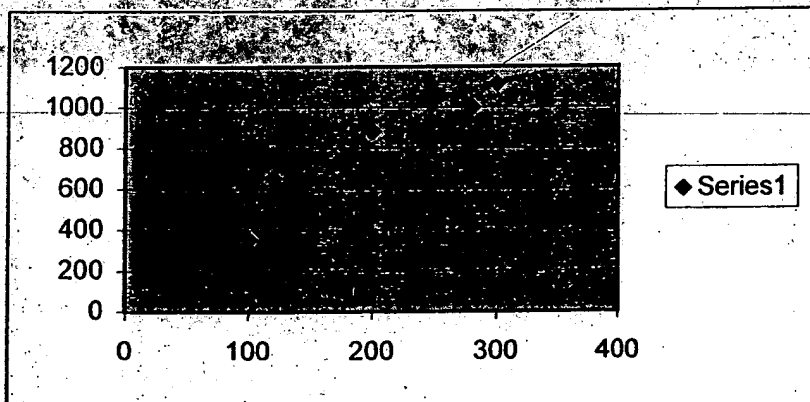
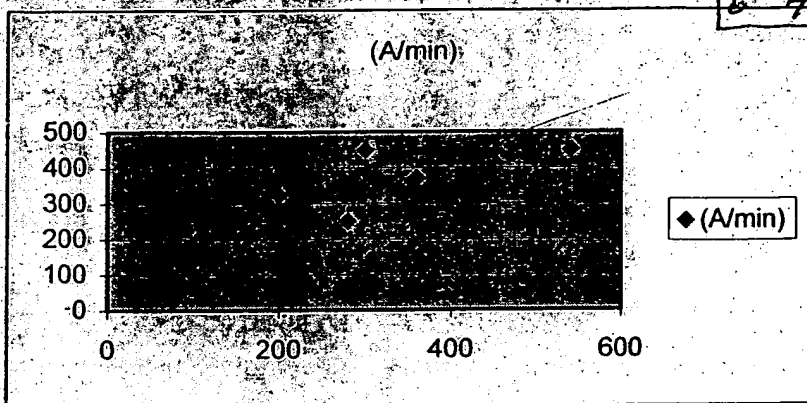
Wfr Slot#	Last polish with	Proc. Conditions	Thickness left	Veeco RMS	AFM RMS		Scrach	
					Center	1/2 radio	Edge	Center
Ir								1/2 radio
Ir21	ZCX200 (pH7)	6 psi/90 rpm	155 nm	7.45 A				Edge
Ir18	CR30+0.2 mol P/A	4 psi/50 rpm	87 nm					
Ir17	ZCX200, pH1.5	4 psi/50 rpm	45 nm		3.16 A	2.70 A	3.11 A	13.7
Ir20	Unproc.			2.71 A	6.80 A	3.43 A	2.63 A	7.09 A
IrO2								18.4
IrO17	ZCX201	6psi/50rpm	84 nm	9.39 A				5.17 A
IrO16	ZCX201	4psi/30rpm	64 nm		1.78 A	1.89 A	2.49 A	
IrO12	CR30+TMAH	4 psi/50 rpm	72 nm	3.10 A	3.10 A	3.06 A	3.28 A	10.2
IrO16	Unproc.	/		3.74 A				5.83 A

d Understood

ire

Date

Wfr type	Abrasive		Chemical		pH	Psi	Platen (rpm)	psi* rpm	RR (A/min)
	Name	Wt%	Name	mol/Kg					
Ir	CR30	2%	PIA-TMAH	0.1	6-7 with TMAH	4	50	200	324
						2	50	100	100
						6	50	300	450
						4	70	280	250
						4	90	360	370
						6	90	540	450
IrO2	CR30	2%	NH2NH2	0.05	9	4	50	200	380
						2	50	100	355
						6	50	300	1116
						4	30	120	650
						4	70	280	1000



$$y = Ax^{\frac{2}{3}} + B$$

$$x^2 + x + x^3 + x^2 + x$$

EXHIBIT

## 8/9/99 Formulation and Process development DOE

Fixed process condition: 4psi/50rpm/51rpm/150ml/min/1-2 min, note only 200 nm thick of Ir or IrO<sub>2</sub>.

## Ir and TEOS wafers polish RR (A/min):

Formula	Chemical	pH	CR30, 0 pass	Abrasive (2 wt%)	CR30, 1 pass	CR30, 2 pass	CR30, 3 pass
	None						
ZCX200	Ir/TEOS						
ZCX206	Ir/TEOS	7					
		7					

1-pass-mth?

IrO<sub>2</sub> and TEOS wafers polish RR (A/min):

Formula	Chemical	pH	CR30, 0 pass	Abrasive (2 wt%)	CR30, 1 pass	CR30, 2 pass	CR30, 3 pass
	None						
ZCX201							
ZCX203		9 to 10					
ZCX203a		11 to 12					
		9 to 10					

3-pass-for-IrO<sub>2</sub>?

## Record the following:

Process observ.: Pad color? Conditionabl? Any corrosion of the tool? Abnormal?

Visual Inspection: Uniform/shining, "break", scratch/crack, finish discoloring?

4-pp: Read mOhms/sq., then convert to thickness with calibration curve. 15 mm edge excl. (120 mm diameter)

Veeco RMS: use 125 mm diameter size.

AFM scratch depth: Do 3 scans at "center", 1/2 radio, and "edge".

## Next DOE:

Pick the best formulation from each group, then

Platen RPM: 50, 70, 90

Down force: 2, 4 psi

Back pressure: range?, try one or two(0.5 and 1 psi ?)

TEOS : Ir/IrO<sub>2</sub> selectivity: 100 : 1 requiredPolish TEOS with all above slurries used for Ir and IrO<sub>2</sub>.

New wfr from Infineon

8/2/99 Box A:

25 wfrs, 200 nm Ir/10 nm Ti/450 nm TEOS/Si. PF939615#01-25.

8/2/99 Box b:

25 wfrs, 200 nm IrO<sub>2</sub>/450 nm TEOS/Si. PF939615#26-50.

8/12/1999, Box1:

15 wfrs, 150 nm Ir/10 nm Ti/350 nm TEOS (patterned, etch depth: 75 nm) on 625 nm thermal oxide on Si. PF937158#11-25.

8/12/1999, Box2:

15 wfrs, 300 nm IrO<sub>2</sub>/350 nm TEOS (patterned, etch depth: 200 nm) on 625 nm thermal oxide on Si. PF937158#36-50.

10 wfrs with denser TEOS layer, 1000 nm TEOS (planar) on Si substrate (reflectometry data enclosed). PF936862#06-15.



Notebook Number: \_\_\_\_\_

## Chemicals and abrasives screening test with Logitech:

Abrasive	Chemical	dip, psi	platen rpm	Flow ml/min	Time, min	Buff, min
2 wt%	0.1 mol/Kg	1.06	40	50	5	2

## In polish: Removal rates (A/min):

Code	Chemical	dip, psi	platen rpm	Flow ml/min	Time, min	Buff, min
Ox1	✓ H <sub>2</sub> O <sub>2</sub> - NH <sub>4</sub> OH to pH 9.8	1.06	40	50	5	2
Ox2						
Ox3						
Ox4						
Ox5						
Ox6						
Ox7						
Ox8						
Re1						
Re2						
Re3						
Re4						
Re5						
Acid1						
Acid2						
Acid3						
Acid4						
Acid5						
Base1						
Base2						

Read and Understood By: \_\_\_\_\_

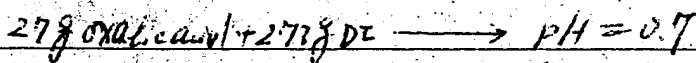
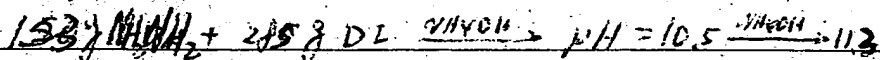
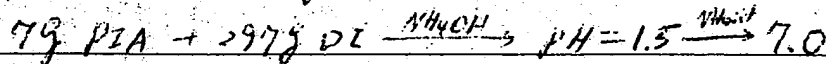
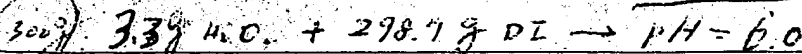
Project:

Notebook Number:

0.1 mol/kg

Meq.

Particle	Chemical	Pressure	PC Size
40 MPa	500 psi	100 PSI	6"
5 min			



May try:  $\text{HNO}_3 + \text{H}_2\text{O}_2$

0.1 mole 0.1 mole

• oxalic acid with other to enhance complexing

Project: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

0.1 ml / 1/3 solute

0.07 ml / 300 g solute. same polish condits; same process condits.

11. Hel 3.0 g + CR30 2<sup>wt%</sup> → 300 g → pH = 0.9 spin fast, <sup>very</sup> black on pad12. CaClO<sub>2</sub> 4.2 g + CR30 2<sup>wt%</sup> → 300 g → pH = 12.5 2 PSI / 2 min with only small 6; see ~~with~~13. CR30, 2<sup>wt%</sup>, blank. black, spin fast.Abrasive screening (List on page 12) : all milled colloidal1. <sup>CR30</sup> 10% x = 2% x 300 g → x = 60 g → pH = 4.3 black on pad

2. VEX300: 10% x = 2% x 300 g → x = 60 g → pH = 5.0 black, spin slowed down

3. CR140: 20% x = 2% x 300 g → x = 30 g → pH = 5.6 black, spin slowed down

4. AL2O<sub>3</sub>: 20% → x = 30 g → pH = 4.0 look like water, spin very slow

5. CMP7000 35% x = 2% x 300 g → x = 97 g → pH = 8.8 (6.4) spin slow

6. CE-100, 10% x ~~filtered, 0.2 μm, pH = 11.0~~ spin very slow  
x = 2% x 300 g → x = 60 g → pH = 11.1



Project: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

perforated IC-1000 pad (3)

IC1400 / K-grooved (6)

• IC1000 / suba 4 stacked

• IC1400 / PSA stacked

## Abrasives:

1. CR30
2. CR6
3. 30% Al <sub>2</sub> O <sub>3</sub> C25 GEL
4. 36.5% Al <sub>2</sub> O <sub>3</sub> C25 GEL
5. 40% SN6-, milled
6. OPSM4 Lot#10678
7. OPSM20, 20% milled
8. D65CR, Lot#8813
9. 24AlO-MgO Lot#8802-CP
10. 12AlO-MgO Lot#9079
11. Al <sub>2</sub> O <sub>3</sub> Lot#10403-3
12. CR85, Lot#8864CR
13
14
15. Nanotek TiO <sub>2</sub> , Lot#17050-015
16. T30CR, C712
17
18
19. Al <sub>2</sub> O <sub>3</sub> Gel C75
20. Cerium/Alumina Lot#10400
21
22. CR1
23. SiC
24. B4C

	heavy	light	thin	up side
lb	30lb	22lb	28.26in <sup>2</sup>	6"

	1.06psi	0.78psi	7.07in <sup>2</sup>	3"
psi	4.24psi	3.11psi		

Head + 9 small ring = 13.970 lb → 1.97 psi

$$R = P \cdot \frac{L}{A}$$

$$R_s = R = \frac{P}{T} \rightarrow P = R_s \cdot T$$

$\uparrow$     $\uparrow$   
 $\frac{\text{lb}}{\text{in}^2}$     $\text{cm, mm}$

R<sub>s</sub> = 1.97R<sub>s</sub>

## EXHIBIT

10

Notebook Number:

Object:

6" pt boats from slot #10 - 25 Unprocessed, on Classroom 4P

#10	154.42	no/min/sq	0.622% cte. dev.	Edge exclusion: 20 mm / 45 pt
#11	161.72		0.789%	20 mm
#12 repeat	166.57		4.707%	5 mm
#11 repeat	163.48		2.608%	10 mm
#10	155.42		1.683%	10 mm
#12	159.81		1.195%	and after
#13	172.93	Ave.	1.879%	
#14	158.22	164	1.634%	
#15	169.41		1.744%	
#16	167.80		1.696%	
#17				
#18				
#19				
#20				
#21				
#22				
#23				
#24				
#25				

Iridium  
recipe

Read and Understood By

Project:

Rt Water CMP

EXHIBIT

tabbler

11

Notebook Number:

4

## 3" Pt on IC1000 pad: (Four Dementation 4-pp)

Wtr I.D.	Date	Abrasive	(wt%)	Chem.	(mol/300g)	pH	mOhms/sq.	Std. Dev. (%)	Thickness (nm)	Delta (nm)	RR (nm/min)
Pt1	8/10/99	Unproc.					355.22	1.38			
Pt2	8/10/99	Unproc.					355.54	1.43			
Pt3	8/10/99	Unproc.					355.54	1.67	Ave.: 357mOhms/sq for 500 nm		
Pt4	8/10/99	Unproc.					356.15	1.28			
Pt5	8/10/99	Unproc.					365.37	1.45			
Pt1-1	8/11/99	None		APS	0.1	1.0	356	1.26	500	0	0
Pt2-1	8/11/99	None		Ca(ClO)2	0.1	12.7	360	100.85	230	270	54
Pt3-1	8/11/99	None		PIA(NH4OH)	0.1	7.0	395	23.92	490	10	2
Pt4-1	8/11/99	None		PIA(TMAH)	0.1	7.0	358	2.3	500	0	0
Pt1-2	8/11/99	None		PIA	0.1	1.1	360	2.3	500	0	0
Pt2-2	8/11/99	None		H2O2(NH4OH)	0.1	10.1	360	79.42	280	0	0
Pt3-2	8/11/99	None		NH2NH2	0.1	10.3	454	33.9	440	50	10
Pt4-2	8/11/99	None		OA+pentandione	0.03 + 0.03	1.1	357	2.45	500	0	0
Pt25-1	8/11/99	None		HNO3+H2O2	0.03 + 0.03	1.0	355	1.36	500	0	0
Pt25-2	8/12/99	None		HCl	0.03	1.0	355	1.41	500	0	0
Pt1-3	8/12/99	CR30	2	HCl	0.03	0.9	can't meas.		center 0	500	>100 center
Pt2-3	8/12/99	CR30	2	None		6 to 7	can't meas.		center 0	500	>250 center
Pt3-3	8/12/99	CR30	2	Ca(ClO)2	0.03	12.5	353	52.53	360	80	40
Six abrasive screening tests below with 2 psi, instead of 4 psi as above:											
Pt3-4	8/13/99	Mill CR65	2	None		4.3	353	23.78	460	0	0
Pt4-3	8/13/99	VIX300	2	None		5	377	8.91	495	5	1
Pt25-3	8/13/99	Mill CR140	2	None		5.6	358	0.88	500	0	0
Pt5-1	8/13/99	AL20	2	None		4	359	1.52	500	0	0
Pt5-1	8/13/99	CMP8000	2	None		8.8	354	1.15	500	0	0
Pt7-1	8/13/99	CE100	2	None		11.1	353	1.03	500	0	0

GR30(10p) NDA updated on 9/20/99

Cerin

26/9/99

Cu mps., after chemistry process

slab # (original)	run	thickness	RMS roughness	code	Comments
{ 1	5000	13065 Å	19.71 Å	Cu1A	Dull circle in
{ 2	5000	11145 Å	27.63 Å	Cu2A	good
{ 3	5100, pH3	13489 Å	23.54 Å	Cu3A	stains
{ 4	"	2629.5 Å	21.99 Å	Cu4A	stains
{ 5	5100, pH5	14211 Å	21.49 Å	Cu5A	good
{ 6	"	14831 Å	19.93	Cu6A	good
{ 7	5100, pH8.5	13140 Å	18.52	Cu7A	2 corrosion spots
{ 8	"	14305 Å	17.96	Cu8A	3 corrosion spots
{ 9	5000 L	13902 Å	18.03		stains/patch
{ 10		14330 Å	18.49		good
{ 11	5000 K	13888 Å	19.76		2 small
{ 12	"	14299 Å	19.13		good

13

Signature

Date

9/29/83

Project: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

IPEC 576 recipe "pt 9-99" (SOP on p47)

2PSC / 200 Rpm / 150 ml. m. / 1 min / Temp 120°C  
18 Rpm (up) part x8

Beaker #	Ref I.D	PSC/Rpm/min	Thickness	Std Dev	RMS (Å)	Comments
#2	Foli # N46M	2/200/1	BPSG 12888 Å	3.73%		FT/50 sec red "dots."
PH3	{ BPSG1-5		[RR = 115 Å/min]			
	pt 11-1	"	pt: 2797.7 Å	6.293%		q-RP
			[RR <sub>pt</sub> = 229 Å/min]			
	pt 11-2	"	pt: 2553.4 Å	6.716%		
			[RR <sub>pt</sub> = 244 Å/min]			
#1	BPSG1-6	2/200/1	12449 Å	3.658%		FT/50 sec red "patches"
PH6.5			[RR = 439 Å/min]			
	pt 11-3	"	pt 2517 Å	6.971%		
			[RR <sub>pt</sub> = 36.4 Å/min]			
	pt 11-4	"	pt 2468 Å	6.856%		
			[RR <sub>pt</sub> = 49 Å/min]			
#3	BPSG1-7	2/200/1	12151 Å	4.441%		
PH=3.0			[RR = 298 Å/min]			
	pt 11-5	"	2301.6 Å			
			[RR <sub>pt</sub> = 166 Å/min]			
#4	BPSG1-8	2/200/1	11919 Å	4.30%		
PH=6.5			[RR = 232 Å/min]			
	pt 11-6	"	2248.3 Å			
			[RR <sub>pt</sub> = 53 Å/min]			

Signature

Date

80

Signature

Date

## EXHIBIT

tabbies

13

object: \_\_\_\_\_

Notebook Number: \_\_\_\_\_

dry  
CR30

chem.  
PLA + etcd NaCl  
CaClO<sub>2</sub>

Micron Pt CMP: IPEC576, Thomas XY, 2psi/200rpm/150ml per min/1 min

Abrasive	Chem	pH	Pt (A/min)	BPSG (A/min)	Pt : BPSG
CR30 (10 p)	PIA(+HCl+NH4OH)	3.0	237	115	2.1
CR30 (10 p)	HDA (+HCl)	6.5	43	439	1.12
CMP3100	PIA(+HCl+NH4OH)	3.0	167	298	1.8
CMP3100	PIA(+HCl+NH4OH)	6.9	53	232	4.4
CR30 (10p)	HDA	8 - 9	91		n/a

From previous <sup>sheet</sup> page:

Micron Pt CMP: AVANTI472, IC1000 K-grooved, 4psi/50 rpm/150mlper min/1min

Abrasive	Chem	Pt (A/min)	Pt roughnes	BPSG (A/min)	BPSG roughne	Pt : BPSG
CR30	HDA <sup>pH 8</sup>	209	20	432	25	1.2
CMP3100	HDA <sup>pH 8.5</sup>	220	29	1075	12	1.5
CR30	PIA <sup>pH 3</sup>	525	11	578	11	1.1
CMP3100	PIA <sup>pH 3.7</sup>	357	8	829	10	1.23

Beaker #

Abrasive

duration

pH

#3 CR30(10p)(152<sup>g</sup>) : 63 g/kg CaClO<sub>2</sub> : 14.3 g/kg (0.1 ml/kg) 12.1

#1 CR30 .. 63 g/kg  
 HDA : 26.4 g/kg (0.4 ml/kg) 8.6  
 Alkal : 2.7 g/kg (0.05 ml/kg)  
 53.4 g

#2 CR30 .. 63 g/kg  
 PIA : 45.6 g/kg (0.2 ml/kg) 1.4 → <sup>+NH4OH</sup> 3.2  
 Alkal : 2.7 g/kg (0.05 ml/kg)  
 NH4OH titrate to pH 2-3

*[Signature]*

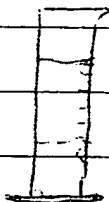
11/23/99

Signature

11/24/99

Date

should start to do pot life, etc. test and improvement.



Time

Settle's level

- Keep #5, #6 overnight.

~~85 setting to 63~~

**Signature**

Date \_\_\_\_\_

94

Date \_\_\_\_\_

**EKC Technology, Inc.**  
**RECAP OF PATENT MATTERS (Updated 11/16/01)**



**Completed Patent Disclosures (1 of 6)**

<b>TITLE:</b>	<b>Composition For Cleaning Chemical-Mechanical Planarization Apparatus</b>	
<b>DOCKET NO.:</b>	20601-pa	
<b>INVENTORS:</b>	Robert Small & Joo-Yun Kim	
<b>STATUS</b>		<b>DATE</b>
Completed Patent Disclosure & Mailed Same To David E. Steuber, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		<b>May 18, 2000</b>

**Completed Patent Disclosures (2 of 6)**

<b>TITLE:</b>	<b>Composition for Tungsten Chemical-Mechanical Planarization Processes</b>	
<b>DOCKET NO.:</b>	20602-pa	
<b>INVENTOR:</b>	Bruce Tredinnick	
<b>STATUS</b>		<b>DATE</b>
Completed Patent Disclosure & Mailed Same To David E. Steuber, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		<b>September 6, 2000</b>

**Completed Patent Disclosures (3 of 6)**

<b>TITLE:</b>	<b>Chemical-Mechanical Planarization Using Ozone</b>	
<b>DOCKET NO.:</b>	20605-pa	
<b>INVENTORS:</b>	Robert Small & Chass Shang	
<b>STATUS</b>		<b>DATE</b>
Completed Patent Disclosure & Mailed Same To David E. Steuber, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		<b>October 19, 2000</b>

**Completed Patent Disclosures (4 of 6)**

<b>TITLE:</b>	<b>Compositions for Chemical-Mechanical Planarization of Copper "Phase I"</b>	
<b>DOCKET NO.:</b>	20604-pa	
<b>INVENTORS:</b>	Robert J. Small, Maria Peterson, Tuan Truong, Mel Carter, Lily Yao	
<b>STATUS</b>		<b>DATE</b>
Completed Patent Disclosure & Mailed Same To George Wolken, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		<b>April 20, 2001</b>



## EKC Technology, Inc.

## RECAP OF PATENT MATTERS (Updated 11/16/01)

## Completed Patent Disclosures (5 of 6)

TITLE:	Compositions for Chemical Mechanical Planarization Of Tantalum and Tantalum Nitride "Phase II"	
DOCKET NO.:	20615-pa	
INVENTORS:	Robert J. Small, Maria Peterson, Tuan Truong, Mel Carter, Lily Yao	
STATUS		DATE
Completed Patent Disclosure & Mailed Same To George Wolken, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		May 30, 2001

## Completed Patent Disclosures (6 of 6)

TITLE:	Compositions for Noble Metal Chemical-Mechanical Planarization Processes	
DOCKET NO.:	20603-pa	
INVENTORS:	Robert Small and Zhefei Chen	
STATUS		DATE
Completed Patent Disclosure & Mailed Same To George Wolken, Esq. @ Skjerven, Morrill, Macpherson, Franklin & Friel		August 22, 2001

## Patent Disclosures In Progress (1 of 2)

TITLE:	Chemical-Mechanical Planarization Using Plastic Particles/Beads	
DOCKET NO.:	20606-pa	
INVENTORS:	Robert Small	
STATUS		DATE
Draft of Patent Disclosure In Progress		Expected Completion Date Is Before November 22, 2001

## Patent Disclosures In Progress (2 of 2)

TITLE:	"Spin Etching of Metal Films"	
DOCKET NO.:	20608-pa	
INVENTORS:	Robert J. Small, Mel K. Carter & Becky Hon	
STATUS		DATE
Draft of Patent Disclosure In Progress		Expected Completion Date Is On or Before December 14, 2001

EXHIBIT

15

**DENNIS A. DEBOO**  
REGISTERED PATENT AGENT

U.S. & INTERNATIONAL PATENT PRACTICE

July 24, 2001

Mr. Robert Small, Ph.D.  
R&D Technical Director  
EKC Technology, Inc.  
2520 Barrington Court  
Hayward, CA 94545

Re: **Draft of Patent Disclosure**

Title: Compositions for Nobel Metal Chemical-Mechanical Planarization Processes

Inventor: Robert Small and Zhefei Chen

Reference No. 20603-pa

Dear Bob:

Enclosed please find a **First** draft of the above-identified patent disclosure.

The enclosed patent disclosure was mainly extracted from Zhefei Chen's Monthly Summary Reports and Excel Spreadsheets. Thus, please **carefully** read the enclosed patent disclosure for technical accuracy.

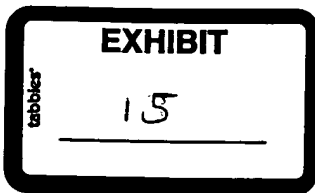
An electronic version of the of the above-identified patent disclosure is also enclosed for your convenience.

Should you have questions, please call.

Respectfully,

*Dennis A. DeBoo*  
Dennis A. DeBoo

Enclosures



**Compositions for Nobel Metal Chemical-Mechanical Planarization Processes**

**FIELD OF THE INVENTION**

The present invention relates generally to compositions for Nobel Metal chemical mechanical planarization or polishing processes and, in particular, to compositions for chemical mechanical planarization or polishing processes of Nobel Metals such as Ir and Pt and their oxides.

**BACKGROUND OF THE INVENTION**

Chemical mechanical planarization (CMP) or polishing processes are well established for modern semiconductor interconnect technology employing metals such as W, Cu and Al. In stark contrast, only very little is known so far about CMP processes of noble metals or noble metal alloys, for example, Au, Ag and the group VIII metals of the periodic table such as Pt and Ir, and their oxides. However, interest in these metals is gaining momentum since they are used as electrodes and barrier materials in Gigabit DRAMs and FeRAMs.

Semiconductor companies, world-wide, are making efforts to commercialize high dielectric constant and ferroelectric thin films in, for example, advanced DRAMs and ferroelectric random access memories (FeRAMs), respectively. These materials include BaSrTiO<sub>3</sub> (BST) for forming capacitors dielectrics (DRAM storage capacitors or coupling capacitors in general circuits) in submicron integrated circuits because of their high dielectric constant. Additionally, materials such as PbZrTiO<sub>3</sub> (PZT) and SrBi<sub>2</sub>Ti<sub>2</sub>O<sub>9</sub> (SBT) that can store charge permanently are employed in the formation of non-volatile FeRAM memory. The chemical properties of BaSrTiO<sub>3</sub>, PbZrTiO<sub>3</sub>, and SrBi<sub>2</sub>Ti<sub>2</sub>O<sub>9</sub> require that they be used with noble metals or noble metal alloys such as Pt, Ir, IrO<sub>2</sub>, et cetera.

However, conventional patterning of these materials by dry etching processes has faced considerable difficulties such as taper angle, fence formation, and residual particles leading to contamination. Some fundamental difficulties of conventional dry etching processes are due to the predominantly physical (not chemical) mechanism for material removal thereby resulting in formation of unwanted structures at the edges of the electrodes.

For the forgoing reasons, there is a need for new, novel and useful chemical compositions for chemical mechanical planarization or polishing processes of Nobel Metal or group VIII metals of the periodic table such as Pt and Ir and their oxides, for example,  $\text{IrO}_2$ . Furthermore, there is a need for new, novel and useful chemical compositions for planarization or polishing of Nobel Metals that are compatible with standard chemical mechanical planarization or polishing equipment. Furthermore, there is a need for CMP slurry compositions that polish Nobel Metals at desired high polishing rates while minimizing surface imperfections, defects, corrosion, recessing and erosion.

## Description

### Ir Polishing Compositions

#### Composition A

One preferred Ir polishing composition pursuant to the present invention ("Composition A") is comprised of an alpha-alumina abrasive, Periodic Acid ( $H_5IO_6$ ), and de-ionized ("DI") water. One example of component concentrations for Composition A is shown in the following table.

#### Composition A Component Concentration

Component	Component Concentration
Alpha-Alumina Abrasive (CR30)	2 wt%
Periodic Acid	0.1mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition A

Thus, for example, 10 kilograms of Composition A requires 200 grams of alpha-alumina abrasive plus 1 mole of Periodic Acid and the remaining amount DI water.

#### pH Ranges

Composition A may have a general pH range of less than or equal to about 2.5 and a preferred pH of about 1.5.

#### Preparation

Generally, Composition A is prepared by first preparing a container of DI water to which the alpha-alumina abrasive (CR30) is added and then the Periodic Acid ( $H_5IO_6$ ) is added.

Composition A is preferably continuously stirred within the container during at least the composition preparation.

CMP Process:

The following table shows one example of the mixing ratio, process, pH and removal rate for Composition A.

Mixing Ratio, Process, pH and Removal Rate

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)
2 wt% Alpha-Alumina Abrasive  0.1mol/1kg Periodic Acid  DI water	4/0/50/51/150	1.5 – 2.5	288

In the above example, the CMP process was carried out on IPEC's AVANTI 472 polisher and was achieved by applying a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed of 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over Suba 4 was used as the polishing pad on the primary platen of the polisher and a polytex pad was used as the buff pad on the secondary platen of the polisher. Composition A was stirred thoroughly before and during its use.

Composition A, when employed according to the above process, provided an Ir removal rate of approximately 288 angstroms per minute.

Additionally, Composition A achieved an Ir removal rate of approximately 375 angstroms per minute when carried out according to the above process with the exception of increasing the down force pressure from 4 psi to a down force pressure of 6 psi. Furthermore, Composition A achieved an Ir removal rate of approximately 400 angstroms per minute when carried out according to the above process with the exception of increasing the table speed from 50 rotations per minute to 70 rotations per minute.

**Ir Polishing Compositions Including Titration with TMAH**

**Composition B**

Another preferred Ir polishing composition pursuant to the present invention ("Composition B") is comprised of an alpha-alumina abrasive, Periodic Acid ( $H_5IO_6$ ), de-ionized ("DI") water, and a base in the form of Tetramethylammonium Hydroxide (TMAH). One example of component concentrations for Composition B is shown in the following table.

**Composition B Component Concentration**

Component	Component Concentration
Alpha-Alumina Abrasive (CR30)	2 wt%
Periodic Acid	0.1mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition B
Tetramethylammonium Hydroxide (TMAH)	Titrate with TMAH to a pH of approximately between 6 and 7

For example, 10 kilograms of Composition B requires 200 grams of alpha-alumina abrasive plus 1 mole of Periodic Acid and the remaining amount DI water. This chemistry is then titrated with TMAH to obtain a final pH of about 6 to about 7.

#### pH Ranges

Composition B may have a general pH range of about 6 to about 7 and a preferred pH of about 7.

#### Preparation

Generally, Composition B is prepared by first preparing a container of DI water to which the alpha-alumina abrasive (CR30) is added. Next, the Periodic Acid ( $H_5IO_6$ ) is added. This chemistry is then titrated with TMAH to obtain a final pH value of about 6 to about 7. Composition B is preferably continuously stirred within the container during at least the composition preparation.

#### CMP Process:

The following table shows one example of the mixing ratio, process, pH and removal rate for Composition B.

#### Mixing Ratio, Process, pH and Removal Rate

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)



2 wt% Alpha-Alumina Abrasive	4/0/50/51/150	6-7	325
0.1mol/1kg Periodic Acid			
DI water			
Titrate with TMAH to a pH of approximately 7			

In the above example, the CMP process was carried out on IPEC's AVANTI 472 polisher and was achieved by applying a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over Suba 4 was used as the polishing pad on the primary platen, and a polytex pad was used as the buff pad on the secondary platen. Composition B was stirred thoroughly before and during its use.

Composition B, when employed according to the above process, provided an Ir removal rate of approximately 325 angstroms per minute.

#### **Ir Polishing Compositions Including Titration with NH<sub>4</sub>OH**

##### **Composition C**

Another preferred Ir polishing composition pursuant to the present invention ("Composition C") is comprised of an alpha-alumina abrasive, Periodic Acid (H<sub>5</sub>IO<sub>6</sub>), de-ionized ("DI") water, and a base in the form of Ammonium Hydroxide (NH<sub>4</sub>OH). One example of component concentrations for Composition C is shown in the following table.

##### **Composition C Component Concentration**

Component	Component Concentration
-----------	-------------------------

Alpha-Alumina Abrasive (CR30)	2 wt%
Periodic Acid	0.1mol/1kg
DI Water	Remaining weight amount to obtain final desired amount of Composition C
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate with NH <sub>4</sub> OH to a pH of approximately 7

For example, 10 kilograms of Composition C requires 200 grams of alpha-alumina abrasive plus 1 mole of Periodic Acid and the remaining amount DI water. This chemistry is then titrated with NH<sub>4</sub>OH to obtain a final pH of about 6 to about 7.

#### pH Ranges

Composition C may have a general pH range of about 6 to about 7 and a preferred pH range of about 7.

#### Preparation

Generally, Composition C is prepared by first preparing a container of DI water to which the alpha-alumina abrasive (CR30) is added. Next, the Periodic Acid (H<sub>5</sub>IO<sub>6</sub>) is added. This chemistry is then titrated with NH<sub>4</sub>OH to obtain a final pH value of about 7. Composition C is preferably continuously stirred within the container during at least the composition preparation.

#### CMP Process:

The following table shows one example of the mixing ratio, process, pH, removal rate and selectivity for Composition C.

Mixing Ratio, Process, pH, Removal Rate, and Selectivity

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)	Ir:TEOS Selectivity
2 wt% Alpha-Alumina Abrasive  0.1mol/1kg Periodic Acid  Remaining % DI water  Titrate with NH <sub>4</sub> OH to a pH of approximately 7	5/0/90/50/150	6 - 7	360	1.8:1

In the above example, the CMP process was carried out on IPEC's AVANTI 472 polisher and was achieved by applying a down force pressure of 5 psi, a back pressure of 0 psi, a table speed of 90 rpm, a carrier speed 50 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over Suba 4 was used as the polishing pad on the primary platen, and a polytex pad was used as the buff pad on the secondary platen. Composition C was stirred thoroughly before and during its use.

Composition C, when employed according to the above process, provided an Ir removal rate of approximately 360 angstroms per minute and an Ir:TEOS selectivity of 1.8:1.

Composition D

Another preferred Ir composition pursuant to the present invention ("Composition D") is comprised of an alpha-alumina abrasive, Periodic Acid (H<sub>5</sub>IO<sub>6</sub>), de-ionized ("DI") water, and a

base in the form of Ammonium Hydroxide ( $\text{NH}_4\text{OH}$ ). One example of component concentrations for Composition D is shown in the following table.

Composition D Component Concentration

Component	Component Concentration
Alpha-Alumina Abrasive (CR30)	2 wt%
Periodic Acid	0.1mol/1kg
DI Water	Remaining weight amount to obtain final desired amount of Composition D
Ammonium Hydroxide ( $\text{NH}_4\text{OH}$ )	Titrate with $\text{NH}_4\text{OH}$ to a pH of approximately 3

For example, 10 kilograms of Composition D requires 200 grams of alpha-alumina abrasive plus 1 mole of Periodic Acid and the remaining amount DI water. This chemistry is then titrated with  $\text{NH}_4\text{OH}$  to obtain a final pH of about 3.

pH Ranges

Composition D may have a general pH range of about 2 to about 4 and a preferred pH range of about 3.

Preparation

Generally, Composition D is prepared by first preparing a container of DI water to which the alpha-alumina abrasive (CR30) is added. Next, the Periodic Acid ( $\text{H}_5\text{IO}_6$ ) is added. This

chemistry is then titrated with  $\text{NH}_4\text{OH}$  to obtain a final pH value of about 3. Composition D is preferably continuously stirred within the container during at least the composition preparation.

CMP Process:

The following table shows one example of the mixing ratio, process, pH, removal rate and selectivity for Composition D.

Mixing Ratio, Process, pH, Removal Rate and Selectivity

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)	Selectivity
2 wt% Alpha-Alumina Abrasive 0.1mol/1kg Periodic Acid Remaining % DI water Titrate with $\text{NH}_4\text{OH}$ to a pH of approximately 3	5/0/90/50/150	3 - 4	320	1:1.5

In the above example, the CMP process was carried out on IPEC's AVANTI 472 polisher and was achieved by applying a down force pressure of 5 psi, a back pressure of 0 psi, a table speed of 90 rpm, a carrier speed 50 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over Suba 4 was used as the polishing pad on the primary platen, and a polytex pad was used as the buff pad on the secondary platen. Composition D was stirred thoroughly before and during its use.

**DENNIS A. DEBOO**  
REGISTERED PATENT AGENT

EXHIBIT

16

U.S. & INTERNATIONAL PATENT PRACTICE

August 22, 2001

George Wolken, Jr., Esq.  
Skjerven, Morrill,  
Macpherson, Franklin  
& Friel  
25 Metro Drive, Suite 700  
San Jose, CA 95110

**RECEIVED**  
BY DOCKET DEPT.

**AUG 23 2001**

SKJERVEN, MORRILL, MACPHERSON,  
LLP.

Re: **Patent Disclosure**

Title: Compositions for Nobel Metal Chemical-Mechanical Planarization Processes

Inventors: Robert J. Small and Zhefei Chen

Our Ref. No.: 20603-pa

Dear George:

Enclosed please find a forty-four page disclosure (also included on disk) for the above referenced invention. Also enclosed are the original supporting data and background material EKC provided for my preparation of this disclosure.

Should you have any questions, please do not hesitate to contact me.

Sincerely,

*Dennis A. DeBoo*  
Dennis A. DeBoo

Enclosures

cc: Mr. Robert Small, Ph.D. (patent disclosure only)

25 Metro Drive  
Suite 700

San Jose  
California 95110

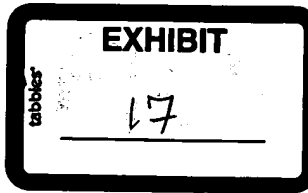
T: 408-453-9200  
F: 408-453-7979

San Francisco, CA  
Austin, TX  
Newport Beach, CA

skjerven morrill  
macpherson LLP

October 1, 2001

Robert Small  
EKC Technology, Inc.  
2520 Barrington Court  
Hayward, CA 94545-1164



Re: U.S. Patent entitled "Compositions for Chemical Mechanical Planarization of Noble Metals, Alloys and Oxides"

Inventors: Robert J. Small; Zhefei Chen

Your Reference: 20603-PA

Our Reference: M-12178 US

Dear Bob:

Enclosed for your review is a draft of the above-identified patent application.

Please review this draft for technical accuracy and completeness of disclosure. We must completely, clearly and accurately describe the invention so as you review this application, please feel free to revise and supplement the description. If there are portions of this draft that are incorrect, please note them. Please also note any blank lines and/or questions located throughout the specification, which must be filled in or answered by you.

It is particularly important that at the time of filing we completely and accurately describe your invention in the application, since we cannot later add new matter to clarify or further describe the invention without running the risk of having to file another application which would lose the benefit of the original filing date for the new matter.

The patent application must:

1. contain a written description of your invention and the manner and process of making and using it;
2. describe your invention in sufficient detail to enable one skilled in the art to make and use your invention; and
3. disclose the best mode contemplated by you of carrying out your invention.

Specifically, if you plan to modify, improve or add features to the invention which are not disclosed in the draft application, these modifications, improvements and features also need to be disclosed in the application as filed. You should also immediately notify us of your planned date for making your invention public (e.g., using your invention publicly in a product, offering for sale a product made using or incorporating your invention, publishing a description of your invention, or providing samples of a product incorporating or made using your invention to a third party).

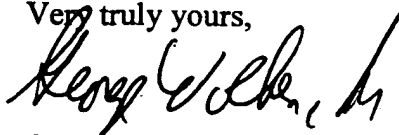
Robert Small  
M-12178 US  
Page 2

Additionally, we must disclose to the United States Patent and Trademark Office all information known by you to be material to the patentability of your invention. Such information is generally any prior art, any existing patents or patent applications relating to or similar to your invention, and any publication, sale, public use, offer to sell, public knowledge, or invention by others of your invention. If you are or become aware of such information, please provide the information to us.

Finally, if you plan to file this application in foreign countries, please note that some countries do not grant priority based on the filing date of a U.S. patent application. If you do not know whether one or more countries in which you intend to file the application grant such priority, please contact us immediately.

Please provide your comments on this application as soon as possible.

Very truly yours,

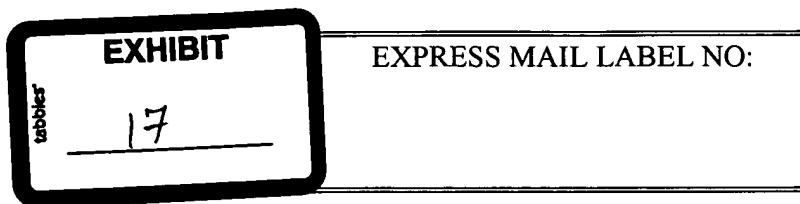
A handwritten signature in dark ink, appearing to read "George Wolken Jr.", with a stylized flourish at the end.

George Wolken Jr.

GW/sr  
Enclosure

806050 v1





**COMPOSITIONS FOR CHEMICAL MECHANICAL PLANARIZATION  
OF NOBLE METALS, NOBLE METAL ALLOYS, NOBLE METAL OXIDES, AND  
COMBINATIONS THEREOF, AND ASSOCIATED METHODS OF USE**

Robert J. Small

Zhefei J. Chen

**BACKGROUND**

**Field of the Invention**

[0001] The present invention relates generally to compositions for chemical mechanical planarization, and more particularly to compositions for chemical mechanical planarization of noble metals, noble metal alloys, noble metal oxides, and combinations thereof, and associated methods of use.

**Description of Related Art**

[0002] Chemical Mechanical Planarization (also referred to as Chemical Mechanical Polishing), or CMP, is commonly used in the manufacture of semiconductor devices and denotes the process of removing material and forming a substantially planar surface before additional layers are deposited and/or additional patterning of the layers occurs. CMP processes have been extensively studied for use in semiconductor fabrication and constitute integral steps in many practical production environments. However, CMP of metals has been studied most extensively in connection with metals such as tungsten, copper, aluminum, tantalum, among others, as well as oxides, nitrides and alloys thereof. See, for example, Chemical Mechanical Planarization of Microelectronic Materials, by J. M. Steigerwald, S. P. Murarka and R. J. Gutmann (John Wiley & Sons 1997), especially Chapters 5 – 8. In contrast, CMP of noble metals, including alloys and oxides thereof, is much less well studied. The term “noble metals” typically refers to less reactive metals such as gold, silver, platinum, iridium and other elements typically found in or near Group VIII of the periodic table.

[0003] Interest in noble metals, and the alloys and oxides thereof, is increasing as such materials are useful as electrode and barrier materials in the fabrication of some electronic devices such as Gigabit ( $10^9$  bit) DRAMs (dynamic random access memories) and FeRAMs (ferroelectric random access memories). Worldwide efforts are underway to commercialize high dielectric constant and ferroelectric thin films for use in capacitive elements as would be applied, for example, in advanced DRAMs and FeRAMs. High dielectric constant materials such as  $\text{BaSrTiO}_3$  (BST) can be used for forming capacitor dielectrics in submicron integrated circuits (*e. g.* in DRAM storage capacitors, coupling capacitors in general circuits, among other uses). Additionally, ferroelectric materials such as  $\text{PbZrTiO}_3$  (PZT) and  $\text{SrBi}_2\text{Ti}_2\text{O}_9$  that can store charge for extended periods of time can be employed in the fabrication of non-volatile FeRAM memory elements. The chemical properties of these (and other) high dielectric constant and ferroelectric materials typically require that they be used in conjunction with noble metals, noble metal oxides and/or noble metal alloys (including Pt, Ir,  $\text{IrO}_2$ , among others). Examples of the use of high dielectric constant and/or ferroelectric materials in semiconductor fabrication and in conjunction with noble metals, noble metal alloys, and noble metal oxides, can be found in the following U.S. Patents: 5,318,927; 5,527,423; 5,976,928; 6,169,305, and references cited therein.

[0004] Conventional patterning of noble metals, noble metal alloys, and noble metal oxides includes the use of dry etching processes. However, dry etching has several disadvantages including unfavorable taper angle, fence formation, and a tendency to produce residual particles leading to contamination. Some of these disadvantages of conventional dry etching are due to the predominantly physical rather than chemical mechanism for material removal. Physical removal of material is prone to the formation of unwanted structures at the edges of the structures being etched (electrodes, for example).

[0005] For the foregoing reasons, among others, there is a need for improved chemical compositions and processes for the CMP of noble metals, noble metal alloys, and/or noble metal oxides including metals from Group VIII of the periodic table, and including in particular, Pt, Ir and  $\text{IrO}_2$ . Furthermore, there is a need for chemical compositions for the CMP of noble metals, noble metal alloys, and/or noble metal oxides that are compatible with standard CMP equipment. There is also a need for CMP compositions that polish noble

metals, noble metal alloys, and/or noble metal oxides at desired high polishing rates while reducing or minimizing surface imperfections, defects, corrosion, recessing and/or erosion.

## SUMMARY

[0006] An exemplary embodiment of the present invention is a composition for chemical mechanical planarization that comprises periodic acid and an abrasive, wherein the periodic acid and the abrasive are present in a combined amount that is sufficient to planarize noble metals, noble metal alloys, noble metal oxides, or any combination thereof. In an embodiment of the composition of the present invention, the periodic acid is present in an amount in a range of from about 0.05 to about 0.3 moles / kilogram, or preferably, from about 0.075 to about 0.175 moles/kilogram, and the abrasive is present in an amount in a range of from about 0.2 to about 6 weight percent, or preferably, from about 0.2 to about 4 weight percent. In another embodiment of the composition of the invention, the abrasive comprises alumina. By way of example, the alumina may be an alpha-alumina, a gamma-alumina, or a combination thereof. In another embodiment, the composition further comprises a sufficient amount of a pH-adjusting agent or titration agent to cause the pH level of the composition to be in a range of from about pH 5 to about pH 9 **[Bob to clarify pH range, particularly as to  $\text{IrO}_2$ ]**, or in a range of from about pH 1 to about pH 4, or preferably, from about pH 2 to about pH 3. In still another embodiment, the composition further comprises a suspension agent, which is preferably a surfactant.

[0007] An exemplary embodiment of a method of the present invention for planarizing a layer on a substrate, where that layer comprises at least one noble metal, noble metal alloy, or noble metal oxide, or a combination thereof. The method comprises providing a composition or slurry comprising periodic acid and an abrasive present in a combined amount that is sufficient to planarize the layer and planarizing the layer with the slurry. In an embodiment of the method, the periodic acid is present in an amount in a range of from about 0.05 to about 0.3 moles/kilogram, or preferably, from about 0.075 to about 0.127 moles/kilogram, and the abrasive is present in an amount in a range of from about 0.2 to about 6 weight percent, or preferably, from about 0.2 to about 4 weight percent. In another embodiment of the method of the invention, the abrasive comprises alumina, such as alpha-alumina, gamma-alumina, or a combination thereof. In other embodiments, the slurry comprises a pH-

adjusting agent or titration agent, such as tetramethyl-ammonium hydroxide or ammonium hydroxide, such that the pH level of the slurry is in a desirable range. In various embodiments the pH range is from about pH 5 to about pH 9 [Bob to clarify], or from about pH 1 to about pH 4 or preferably from about pH 2 to about pH 3.

**[INSERT PARAGRAPH DEFINING PLANARIZATION ONCE PROVIDED BY BOB: Paragraph should set forth how it is determined if something is “planarized”, such as by testing for uniformity across the wafer (what degree of uniformity is sufficient / good?), testing properties of a metal feature on the wafer surface (what test is used; what result is sufficient/ good?), testing for dishing/erosion (what test is used; what amount of dishing / erosion can be tolerated?).]**

**[Make sure BEST MODE covered herein.]**

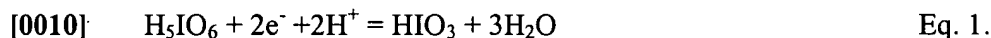
## **DETAILED DESCRIPTION**

[0008] In the following examples, all numerical values and ranges are approximate unless explicitly stated otherwise.

### **Ir Polishing Compositions**

#### **Composition A**

[0009] A polishing composition (such as “Composition A”) useful for polishing iridium pursuant to some embodiments of the present invention is comprised of an abrasive (typically an alumina) and periodic acid ( $\text{H}_5\text{IO}_6$ ) in aqueous solution (advantageously in distilled or deionized water, referred to collectively herein as “DI” water). Periodic acid is capable of participating in a fairly complex group of chemical reactions. Periodic acid is a rather weak acid ( $K_a \approx 5.1 \times 10^{-5}$ ) and a strong oxidizing agent under acidic conditions ( $E^\circ = 1.6 \text{ V}$ ). Depending on the pH of the medium containing periodic acid, different reactive species can be called into play including  $\text{H}_5\text{IO}_6$ ,  $\text{H}^+$ ,  $\text{H}_4\text{IO}_6^-$ ,  $\text{IO}_4^-$ ,  $\text{H}_3\text{IO}_6^{2-}$ . During the short contact time in typical CMP processing, the primary periodic acid reaction is thought to be that represented below in Equation 1 (Eq. 1).



[0011] The reaction represented in Equation 1 is believed to be the primary CMP reaction involving periodic acid, although additional or different reactions may participate within the scope of the present invention.

[0012] Various CMP compositions are described herein in terms of the reactants and other chemical components that are mixed or otherwise combined to form the desired CMP slurry. However, it is recognized that a complex set of chemical processes typically follows blending of the CMP components that may destroy or alter, entirely or in part, one or more of the blended components. The CMP solutions comprising some embodiments of the present invention are described herein in terms of the blended components, with the understanding that the chemical composition (or range of compositions) of the resulting CMP slurry is the necessary result of chemical processes occurring between and among the blended components under the conditions specified. Thus, descriptions herein of the components blended to form a CMP slurry are intended to encompass the chemical species resulting from such blending under the condition (or set of conditions) specified.

[0013] One example of component concentrations for Composition A is shown in the following Table I.

Composition A Component Concentration

**Table I: Typical Composition A**

Component	Component Concentration
Alumina Abrasive	2 weight % ("wt %")
Periodic Acid	0.1 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition A

[0014] Thus, for example, a 10 kilogram mixture of Composition A may be prepared by combining 200 grams of an alumina (whether alpha-, gamma-, or a combination of alpha- and gamma-alumina) abrasive, 1 mole of periodic acid and the remaining of amount DI water. One form of alpha-alumina abrasive advantageously used in connection with some

compositions herein is the commercial product "CR-30" manufactured by Baikowski Chimie Co. of Annacey Cedex 9, France. Other sources of alpha-alumina, as well as sources of gamma-alumina or alpha- and gamma-alumina, may also be utilized.

#### pH Ranges

[0015] Composition A typically has a pH range from about 1 to about 2.5, and favorably (in terms of performance), a pH of about 1.5.

#### Preparation

[0016] Generally, Composition A is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and subsequently adding the periodic acid ( $\text{H}_5\text{IO}_6$ ). The composition is typically stirred continuously within the container during at least the time of the preparation of the composition.

#### CMP Process

[0017] A typical example of the mixing ratio, process, pH and removal rate associated with Composition A is set forth in Table A, in which "A/min" denotes Angstroms ( $10^{-10}$  meter) of material removed per minute of processing.

#### Mixing Ratio, Process, pH and Removal Rate

**Table A**

<b>Mixing Ratio</b>	<b>Process</b>	<b>pH</b>	<b>Ir Removal Rate (A/min)</b>
2 wt % Alpha-Alumina Abrasive	4/0/50/51/150	1.5 – 2.5	288
0.1 mol/l kg Periodic Acid			
DI Water			

[0018] In the example of Table A, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed of 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen of the

polisher, and a Polytex pad was used as the buff pad on the secondary platen of the polisher. Composition A was stirred thoroughly before and during its use.

[0019] When employed according to the process of Table A, Composition A provided an Ir removal rate of approximately 288 Angstroms per minute. Additionally, Composition A provided an Ir removal rate of approximately 375 Angstroms per minute when carried out according to the above-described process (as set forth in Table A) with one variation, namely, applying a higher down force pressure of 6 psi. Furthermore, Composition A provided an Ir removal rate of approximately 400 Angstroms per minute when carried out according to the process set forth in Table A with one variation, namely, applying a higher table speed of 70 rpm. Generally, high removal rates (in terms of the material targeted for removal) are preferred.

#### **Ir Polishing Compositions Including Titration with TMAH**

##### **Composition B**

[0020] Other Ir polishing compositions (such as "Composition B") pursuant to some embodiments of the present invention comprise an alumina abrasive (alpha-, gamma-, or both), periodic acid ( $\text{H}_5\text{IO}_6$ ), DI water, and a pH-adjusting agent, or a base, typically tetramethylammonium hydroxide (TMAH). One example of component concentrations for Composition B is shown in Table II.

##### **Composition B Component Concentration**

**Table II: Typical Composition B**

Component	Component Concentration
Alpha-Alumina Abrasive	2 wt %
Periodic Acid	0.1 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition B
Tetramethylammonium Hydroxide (TMAH)	Titrate with TMAH to a pH of approximately between 6 and 7

[0021] By way of example, a 10 kilogram mixture of Composition B may be prepared by combining 200 grams of an alumina abrasive, 1 mole of periodic acid and the remaining amount of DI water. This mixture is then titrated with the titration agent TMAH to obtain a final pH of about 6 to about 7.

#### pH Ranges

[0022] Composition B typically has a pH range from about 6 to about 7 and, advantageously (in terms of performance), a pH of about 7.

#### Preparation

[0023] Generally, Composition B is prepared by adding the alumina abrasive to a container of DI water and subsequently adding the periodic acid ( $\text{H}_5\text{IO}_6$ ). This mixture is then titrated with TMAH to obtain a final pH value of about 6 to about 7. Composition B is advantageously continuously stirred within the container during at least the period of composition preparation.

#### CMP Process

[0024] An example of the mixing ratio, process, pH and removal rate associated with Composition B is set forth in Table B.

#### Mixing Ratio, Process, pH and Removal Rate

**Table B**

<b>Mixing Ratio</b>	<b>Process</b>	<b>pH</b>	<b>Ir Removal Rate (A/min)</b>
2 wt % Alpha-Alumina Abrasive	4/0/50/51/150	6-7	325
0.1 mol/1 kg Periodic Acid			
DI Water			
Titrate with TMAH to a pH of approximately 7			



[0025] In the example of Table B, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen, and a Polytex pad was used as the buff pad on the secondary platen. Composition B was stirred thoroughly before and during its use.

[0026] When employed according to the above-described process, Composition B provided an Ir removal rate of approximately 325 Angstroms per minute.

#### **Ir Polishing Compositions Including Titration with $\text{NH}_4\text{OH}$**

##### **Composition C**

[0027] Other Ir polishing compositions (such as "Composition C") pursuant to some embodiments of the present invention are comprised of an alumina (alpha-, gamma-, or both) abrasive, periodic acid ( $\text{H}_5\text{IO}_6$ ), DI water, and a pH-adjusting agent or a base, such as ammonium hydroxide ( $\text{NH}_4\text{OH}$ ). One example of component concentrations for Composition C is shown below in Table III.

##### **Composition C Component Concentration**

**Table III: Typical Composition C**

Component	Component Concentration
Alpha-Alumina Abrasive	2 wt %
Periodic Acid	0.1 mol/1 kg
DI Water	Remaining weight amount to obtain final desired amount of Composition C
Ammonium Hydroxide ( $\text{NH}_4\text{OH}$ )	Titrate with $\text{NH}_4\text{OH}$ to a pH of approximately 7

[0028] By way of example, 200 grams of an alumina abrasive, 1 mole of periodic acid and the remaining amount of DI water may be combined to provide a 10 kilogram mixture of

Composition C. This mixture is then titrated with titration agent  $\text{NH}_4\text{OH}$  to obtain a final pH of about 6 to about 7.

#### pH Ranges

[0029] Composition C typically has a pH range from about 6 to about 7 and a favorable pH of about 7.

#### Preparation

[0030] Generally, Composition C is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and subsequently adding the periodic acid ( $\text{H}_5\text{IO}_6$ ). This mixture is then titrated with the titration agent  $\text{NH}_4\text{OH}$  to obtain a final pH value of about 7. Composition C is advantageously stirred continuously within the container during at least the period of composition preparation.

#### CMP Process

[0031] An example of the mixing ratio, process, pH, removal rate and selectivity associated with Composition C is set forth in Table C.

#### Mixing Ratio, Process, pH, Removal Rate, and Selectivity

**Table C**

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)	Ir:TEOS Selectivity
2 wt % Alpha-Alumina Abrasive	5/0/90/50/150	6 - 7	360	1.8:1
0.1 mol/l kg Periodic Acid				
Remaining % DI Water				
Titrate with $\text{NH}_4\text{OH}$ to a pH of approximately 7				

[0032] In the example of Table C, the CMP process was carried out using an IPEC 472

polisher and employing a down force pressure of 5 psi, a back pressure of 0 psi, a table speed of 90 rpm, a carrier speed 50 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen, and a Polytex pad was used as the buff pad on the secondary platen. Composition C was stirred thoroughly before and during its use.

[0033] When employed according to the process of Table C, Composition C provided an Ir removal rate of approximately 360 Angstroms per minute. The Ir removal rates were compared with tetraethoxysilane (TEOS) removal rates, yielding a Ir:TEOS selectivity of 1.8:1. Generally speaking, high selectivity ratios (in terms of the material targeted for removal to another material) are preferred.

#### Composition D

[0034] According to some embodiments of the present invention, other Ir polishing compositions (such as "Composition D") are comprised of an alumina (alpha-, gamma-, or both) abrasive, periodic acid ( $H_5IO_6$ ), DI water and a pH-adjusting agent or base such as ammonium hydroxide ( $NH_4OH$ ). Illustrative component concentrations for Composition D are shown in the following Table IV.

#### Composition D Component Concentration

**Table IV: Typical Composition D**

Component	Component Concentration
Alpha-Alumina Abrasive	2 wt %
Periodic Acid	0.1 mol/1 kg
DI Water	Remaining weight amount to obtain final desired amount of Composition D
Ammonium Hydroxide ( $NH_4OH$ )	Titrate with $NH_4OH$ to a pH of approximately 3

[0035] By way of example, 200 grams of alumina abrasive, 1 mole of periodic acid and the

remaining amount DI water may be combined to provide a 10 kilogram mixture of Composition D. This mixture is then titrated with titration agent  $\text{NH}_4\text{OH}$  to obtain a final pH of about 3.

#### pH Ranges

[0036] Composition D typically has a pH range from about 2 to about 4 and a favorable pH value of about 3.

#### Preparation

[0037] Generally, Composition D is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and subsequently adding the periodic acid ( $\text{H}_5\text{IO}_6$ ). This mixture is then titrated with  $\text{NH}_4\text{OH}$  to obtain a final pH value of about 3. Composition D is favorably continuously stirred within the container during at least the time of composition preparation.

#### CMP Process

[0038] One example of the mixing ratio, process, pH, removal rate and Ir:TEOS selectivity associated with Composition D is set forth in Table D.

#### Mixing Ratio, Process, pH, Removal Rate and Selectivity

**Table D**

Mixing Ratio	Process	pH	Ir Removal Rate (A/min)	(Ir:TEOS) Selectivity
2 wt % Alpha-Alumina Abrasive	5/0/90/50/150	3 - 4	320	1:1.5
0.1 mol/l kg Periodic Acid				
Remaining % DI Water				
Titrate with $\text{NH}_4\text{OH}$ to a pH of approximately 3				

[0039] In the example set forth in Table D, the CMP process was carried out using an IPEC

472 polisher and employing a down force pressure of 5 psi, a back pressure of 0 psi, a table speed of 90 rpm, a carrier speed 50 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen, and a Polytex pad was used as the buff pad on the secondary platen. Composition D was stirred thoroughly before and during its use.

[0040] When employed according to the above-described process (as set forth in Table D), Composition D provided an Ir removal rate of approximately 320 Angstroms per minute. The Ir removal rates were compared with tetraethoxysilane (TEOS) removal rates, yielding a Ir:TEOS selectivity of 1:1.5.

#### **Ir Polishing Compositions including Suspension Agents**

[0041] Other examples of Ir polishing compositions pursuant to some embodiments of the present invention comprise one or more agents for making an improved suspension. Typically such suspension-improving agents (hereinafter, "suspension agents") include abrasives.

#### **Composition E**

[0042] For example, some such Ir polishing compositions (such as "Composition E") comprise the components of Composition D and a second abrasive as a suspension agent. By way of example, in some such Ir polishing compositions the second abrasive may be Alumina-C as a 15% suspension. [Bob to provide source of Alumina-C.] One example of component concentrations for Composition E is set forth in Table V.

Composition E Component Concentration

**Table V: Typical Composition E**

Component	Component Concentration
Alpha-Alumina Abrasive	2 wt %
Periodic Acid	0.1 mol/l kg
DI Water	Remaining weight amount to obtain final desired amount of

	Composition E
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate with NH <sub>4</sub> OH to a pH of approximately 3
Alumina-C (15% suspension)	0.9 wt %

#### pH Ranges

[0043] Composition E typically has a pH range from about 2 to about 4 and, advantageously, a pH of about 3.

#### Preparation

[0044] Generally, Composition E is prepared by adding the alpha-alumina abrasive (such as CR-30) to a container of DI water and subsequently adding the periodic acid (H<sub>5</sub>IO<sub>6</sub>). This mixture is then titrated with NH<sub>4</sub>OH to obtain a final pH value of about 3. Finally, the second abrasive is added. Continuous stirring is maintained during at least the period of composition preparation.

#### CMP Process

[0045] An example of the mixing ratio, process, pH, removal rate and selectivity associated with Composition E is set forth in Table E.

#### Mixing Ratio, Process, pH, Removal Rate, and Selectivity

**Table E**

Mixing Ratio	Process	pH	Ir Removal Rate (Å/min)	(Ir:TEOS) Selectivity

2 wt % Alpha-Alumina Abrasive	5/0/90/50/150	3 - 4	260	1:2.2
0.1 mol/l kg Periodic Acid				
Remaining % DI Water				
Titrate with NH <sub>4</sub> OH to a pH of approximately 3				
0.9 wt % Second Abrasive (e.g., Alumina-C)				

[0046] In the example set forth in Table E, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 5 psi, a back pressure of 0 psi, a table speed of 90 rpm, a carrier speed of 50 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as polishing pad on the primary platen, and a Polytex pad was used as the buff pad on the secondary platen. Composition E was stirred thoroughly before and during its use.

[0047] When employed according to the process of Table E, Composition E provided an Ir removal rate of approximately 260 Angstroms per minute. The Ir removal rates were compared with tetraethoxysilane (TEOS) removal rates, yielding a Ir:TEOS selectivity of 1:2.2.

**[FOR COMPOSITIONS F-K, Bob to provide the following: (1) periodic acid concentration in moles/kilogram; and (2) abrasive concentration in weight percent relative to the composition.]**

#### Composition F

[0048] Other Ir polishing compositions (such as "Composition F") pursuant to the some embodiments of the present invention comprise the components of Composition C and a second abrasive as a suspension agent. In one such composition, a slurry suspension agent, Laponite (a product of Southwestern Clay Co. of Gonzales, Texas) was used. Laponite is typically hydrous sodium lithium magnesium fluoro-silicate (Laponite B), hydrous sodium lithium magnesium silicate (Laponite D, RD, ED, HB, G, XLG), hydrous sodium lithium magnesium silicate modified with tetra sodium pyrophosphate (Laponite DS, RDS, XLS, S,

JS, MS), or hydrous sodium lithium magnesium silicate treated to give a fluoride loading of 2000 ppm (Laponite DF). Although any of the types of Laponite can be used with comparable results, Laponite B was used in the example described below.

[0049] One example of component concentrations for Composition F is shown in Table VI.

Composition F Component Concentration

**Table VI: Typical Composition F**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	8 grams
Laponite (Second Abrasive)	0.5 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %) (First Abrasive)	12.5 grams

Preparation

[0050] In one example, Composition F is prepared by combining an “Oxidizer A”, described below, and an “Abrasive A”, also described below. In the preparation of Oxidizer A, the periodic acid (H<sub>5</sub>IO<sub>6</sub>) is added to a container of DI water (76 grams). This mixture is then titrated with pH-adjusting agent or titration agent NH<sub>4</sub>OH to a final pH value of about 7. This resultant mixture is referred to herein as Oxidizer A. Abrasive A is prepared by adding the Laponite and the alumina abrasive to eight grams of DI water. Oxidizer A is added to Abrasive A to produce Composition F. Continuous stirring is maintained during at least the period of composition preparation.



### Composition G

[0051] Other Ir polishing compositions (such as “Composition G”) pursuant to some embodiments of the present invention comprise the components of Composition C and a slurry suspension agent, such as the surfactant Darvan C. Darvan C is a commercial ammonium polymethacrylate aqueous solution sold by R. T. Vanderbilt Company, Inc. of Norwalk, CT.

[0052] One example of component concentrations for Composition G is set forth in Table VII.

### Composition G Component Concentration

**Table VII: Typical Composition G.**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	8 grams
Darvan C	0.5 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %) (First Abrasive)	12.5 grams

### Preparation

[0053] In one example, Composition G is prepared by combining Oxidizer A and “Abrasive B”, as described below. Oxidizer A is prepared as previously described in relation to Composition F. Abrasive B is prepared by adding the Darvan C and the alumina abrasive to eight grams of DI water. Oxidizer A is added to Abrasive B to produce Composition G. Continuous stirring is maintained during the composition preparation.

### Composition H

[0054] Other Ir polishing compositions (such as “Composition H”) pursuant to some embodiments of the present invention comprise the components of Composition C and a second abrasive as a suspension agent. In some embodiments, the second abrasive is LUDOX TM-50. LUDOX TM-50 is a commercial colloidal silica abrasive of E. I. Du Pont de Nemours and Company, having advantageous properties in terms of particle size and contribution to composition suspension and stability.

[0055] One example of component concentrations for Composition H is set forth in Table VIII.

### Composition H Component Concentration

**Table VIII: Typical Composition H**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	8 grams
LUDOX TM-50 (Second Abrasive)	0.5 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %) (First Abrasive)	12.5 grams

### Preparation

[0056] Generally, Composition H is prepared by combining Oxidizer A and “Abrasive C”, as described below. Oxidizer A is prepared as previously described. Abrasive C is prepared by adding the LUDOX TM-50 (the second abrasive) and the alumina abrasive (the first abrasive) to eight grams of DI water. Oxidizer A is added to Abrasive C to produce Composition H.

Continuous stirring is maintained during at least the composition preparation.

#### Composition I

[0057] Other Ir polishing compositions (such as “Composition I”) pursuant to some embodiments of the present invention comprise the components of Composition C and a slurry suspension agent. In some embodiments, the suspension agent is ethyl carbonate. One example of component concentrations for Composition I is set forth in Table IX.

#### Composition I Component Concentration

**Table IX: Typical Composition I**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	8 grams
Ethyl Carbonate	0.5 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %)	12.5 grams

#### Preparation

[0058] In one example, Composition I is prepared by combining Oxidizer A and “Abrasive D”, as described below. Oxidizer A is prepared as previously described. Abrasive D is prepared by adding the ethyl carbonate and the alumina abrasive to eight grams of DI water. Oxidizer A is added to Abrasive D to produce Composition I. Continuous stirring is maintained during the composition preparation.

#### Composition J

[0059] Other Ir polishing compositions (such as “Composition J”) pursuant to some embodiments of the present invention comprise the components of Composition C and an

organic acid as a suspension agent. In one embodiment, the organic acid is succinic acid. In other embodiments, alternative water soluble organic acids (e.g. mono-, di-, and tri-functional acids) can be used, as can other suspension agents or surfactants that act to suspend the abrasive. One example of component concentrations for Composition J is set forth in Table X.

Composition J Component Concentration

**Table X: Typical Composition J**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	7.5 grams
Succinic acid	1.0 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %)	12.5 grams

Preparation

[0060] In one example, Composition J is prepared by combining Oxidizer A and “Abrasive E”. Oxidizer A is prepared as previously described. Abrasive E is prepared by adding the succinic acid (as an exemplary organic acid) and the alumina abrasive to 7.5 grams of DI water. Oxidizer A is added to Abrasive E to produce Composition J. Continuous stirring is maintained during the composition preparation.

Composition K

[0061] Other Ir polishing compositions (such as “Composition K”) pursuant to the present invention comprise the components of Composition C and a second abrasive as a suspension agent. In some embodiments, the second abrasive is an alumina abrasive in the form of “CR-

140". Cr-140 is a commercial abrasive product manufactured by Baikowski Chimie Co. of Annacey Cedex 9, France, believed to comprise about 95% gamma-alumina and about 5% alpha-alumina. One example of component concentrations for Composition K is set forth in the Table XI.

Composition K Component Concentration

**Table XI: Typical Composition K**

Component	Component Concentration
Periodic Acid	2.3 grams
DI Water	76 grams
Ammonium Hydroxide (NH <sub>4</sub> OH)	Titrate the above components with NH <sub>4</sub> OH to a pH of approximately 7
DI Water	3.5 grams
CR-140 @ 20 wt % (Second Abrasive)	5.0 grams
Alpha-Alumina Abrasive (CR-30 @ 16 wt %) (First Abrasive)	12.5 grams

Preparation

[0062] In one example, Composition K is prepared by combining Oxidizer A (prepared as previously described) and "Abrasive F". Abrasive F is prepared by adding CR-140 (the second abrasive) and CR-30 (the first abrasive) to 3.5 grams of DI water. Oxidizer A is added to Abrasive F to produce Composition K. Continuous stirring is maintained during the composition preparation.

[0063] Preparation conditions associated with Compositions F through K are set forth below in Table 1.

Compositions F Through K Preparation Conditions

**Table 1**

Composition	DI Water	Second Abrasive	First Abrasive	Stir Time	Oxidizer	Stir Time
Composition F	8 grams	0.5 grams Laponite	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.
Composition G	8 grams	0.5 grams Darvan C	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.
Composition H	8 grams	0.5 grams LUDOX TM-50	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.
Composition I	8 grams	0.5 grams Ethyl Carbonate	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.
Composition J	7.5 grams	1.0 grams Succinic acid	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.
Composition K	3.5 grams	5 grams CR-140 (20% wt)	12.5 grams	2 hours	79 grams of Oxidizer A	> 20 min.

[0064] Table 2 below sets forth the pH, settling time, Ir removal rate and Ir:TEOS selectivity associated with Compositions F through K. As used herein, settling time refers to the time it takes for a homogenous slurry mixture to settle in an ambient environment so that a clear top layer is formed. In these examples, the unit of measure for settling time is millimeters of clear liquid (i.e., the depth of the clear top layer measured from the top of the original homogenous mixture) in a given unit of time. For instance, a settling time of 9 mm/10 min indicates that a 9 mm deep layer of clear liquid was formed after 10 minutes of standing in an ambient environment. Generally, a suitable slurry, such as any of the examples of Compositions F through K, should not settle in a relatively “hard” or packed manner such that the slurry can’t be resuspended with minimal agitation.

pH, Settling Time, Ir Removal Rate and Selectivity for Compositions F Through K

**Table 2**

Composition	PH	Settling Time (mm/min)	Ir Removal Rate (A/min)	(Ir:TEOS) Selectivity
Composition F	7.3	9 mm/10 min	240	2.7:1
Composition G	7.3	9 mm/10 min	340	2.1:1
Composition H	7.3	9 mm/10 min	240	3.9:1
Composition I	7.3	9 mm/10 min	350	2.3:1
Composition J	6.7	5 mm/2 hrs	80	1:1.3
Composition K	6.9	10 mm/10 min	230	3.4:1

[0065] Each of the CMP processes set forth in Table 2 above was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Compositions F through K were stirred thoroughly before and during their use.

### **IrO<sub>2</sub> Polishing Compositions**

#### **Composition L (for polishing IrO<sub>2</sub>)**

[0066] Iridium oxide (IrO<sub>2</sub>) polishing compositions (such as “Composition L”) pursuant to some embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, hydrazine hydrate (NH<sub>2</sub>-NH<sub>2</sub>·H<sub>2</sub>O), and DI water. It is believed that the caustic hydrazine hydrate serves as a reducing agent. An example of component concentrations for Composition L is set forth below in Table XII.

#### **Composition L Component Concentration**

**Table XII: Typical Composition L**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Hydrazine hydrate	0.1 mol/l kg

DI Water	Remaining weight amount to obtain final desired amount of Composition L
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#### pH Ranges

[0067] Composition L typically has pH range from about 8 to about 10 and, advantageously, a pH range from about 9.0 to about 9.5.

#### Preparation

[0068] Generally, Composition L is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and subsequently adding the hydrazine hydrate. Composition L is typically continuously stirred within the container during at least the period of composition preparation.

[0069] An example of the mixing ratio, process, pH and removal rate associated with Composition L is set forth below in Table L.

CMP Process {**RICH** -- please reformat so that this heading precedes the paragraph directly above.}

#### Mixing Ratio, Process, pH and Removal Rate

**Table L**

Mixing Ratio	Process	pH	IrO <sub>2</sub> Removal Rate (A/min)
2 wt % Alpha-Alumina Abrasive	4/0/50/51/150	9.0-9.5	880
0.1 mol/l kg Hydrazine hydrate			
DI Water			

[0070] In the example set forth in Table L, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen of the polisher, and a Polytex pad was used as buff pad on the secondary platen of the



polisher. Composition L was stirred thoroughly before and during its use.

[0071] When employed according to the process set forth in Table L, Composition L provided an IrO<sub>2</sub> removal rate of approximately 880 Angstroms per minute.

[0072] Another example of component concentrations for Composition L (denoted as "Composition L(a)") is shown below in Table XIIa.

Composition L(a) Component Concentration

**Table XIIa: Typical Composition L(a) (additional embodiments)**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Hydrazine hydrate	0.05 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition L

pH Ranges

[0073] The example of Table XIIa has a favorable pH value of about 9.0.

CMP Process

[0074] An example of the mixing ratio, process, pH and removal rate associated with Composition L(a) is set forth below in Table L(a).

[0075] Mixing Ratio, Process, pH and Removal Rate {RICH -- Please reformat to remove the paragraph # indication here. This should be a heading, not a numbered paragraph.}

**Table L(a)**

Mixing Ratio	Process	pH	IrO <sub>2</sub> Removal Rate (A/min)
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2 wt % Alpha-Alumina Abrasive	4/0/50/51/150	9.0	740
0.05 mol/l kg Hydrazine hydrate			
DI Water			

[0076] In the example set forth in Table L(a), the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed of 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen of the polisher, and a Polytex pad was used as buff pad on the secondary platen of the polisher. The above Composition L(a) was stirred thoroughly before and during its use.

[0077] When employed according to the process of Table L(a), Composition L(a) provided an IrO<sub>2</sub> removal rate of approximately 740 Angstroms per minute.

#### Composition M (for polishing IrO<sub>2</sub>)

[0078] Other iridium oxide (IrO<sub>2</sub>) polishing compositions (such as "Composition M") pursuant to some embodiments of the present invention are comprised of an alumina (alpha-, gamma, or both) abrasive, tetramethylammonium hydroxide (TMAH), and DI water. An example of component concentrations for Composition M is set forth below in Table XIII.

#### Composition M Component Concentration

**Table XIII: Typical Composition M**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Tetramethylammonium Hydroxide	0.1 mol/l kg
DI Water	Remaining weight amount to obtain final desired amount of Composition M

### pH Ranges

[0079] Composition M typically has a pH range from about 9 to about 11 and, favorably, a pH of about 10.

### Preparation

[0080] Generally, Composition M is prepared by adding the alpha-alumina abrasive (CR-30) to a container of DI water and subsequently adding tetramethylammonium hydroxide (TMAH). Composition M is preferably continuously stirred within the container during at least the composition preparation.

[0081] CMP Process {RICH -- please reformat so that this heading has no associated paragraph number.}

[0082] An example of the mixing ratio, process, pH and removal rate associated with Composition M is set forth below in Table M.

### Mixing Ratio, Process, pH and Removal Rate

**Table M**

Mixing Ratio	Process	pH	IrO <sub>2</sub> Removal Rate (A/min)
2 wt % Alpha-Alumina Abrasive 0.1 mol/kg Tetramethylammonium Hydroxide DI Water	4/0/50/51/150	10	635

[0083] In the example set forth in Table M, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed of 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen of the polisher, and a Polytex pad was used as buff pad on the secondary platen of the polisher. The Composition M of the example was stirred thoroughly before and during its use.

[0084] When employed according to the above process, Composition M provided an IrO<sub>2</sub>.

removal rate of approximately 635 Angstroms per minute.

[0085] Another example of component concentrations for Composition M (denoted herein as “Composition M(a)”) is set forth below in Table XIIIa.

Composition M(a) Component Concentration

**Table XIIIa: Typical Composition M(a)**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Tetramethylammonium Hydroxide	0.03 mol/1 kg
DI Water	Remaining weight amount to obtain final desired amount of Composition M

pH Ranges

[0086] Composition M(a) has an advantageous pH range from about 9 to about 10.

CMP Process

[0087] An example of the mixing ratio, process, pH and removal rate for Composition M(a) is set forth below in Table M(a).

Mixing Ratio, Process and Removal Rate

**Table M(a)**

Mixing Ratio	Process	pH	IrO <sub>2</sub> Removal Rate (A/min)
2 wt % Alpha-Alumina Abrasive	4/0/50/51/150	9 - 10	320
0.03 mol/kg Tetramethylammonium Hydroxide			
DI water			

[0088] In the example set forth in Table M(a), the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed of 51 rpm, and a composition flow rate of 150 ml/min. A stacked pad of IC-1000 over a Suba IV was used as the polishing pad on the primary platen of the polisher, and a Polytex pad was used as buff pad on the secondary platen of the polisher. The Composition M(a) of this example was stirred thoroughly before and during its use.

[0089] When employed according to the process of Table M(a), Composition M(a) provided an  $\text{IrO}_2$  removal rate of approximately 320 Angstroms per minute.

### **Platinum (Pt) Polishing Compositions**

#### **Composition N**

[0090] Platinum (Pt) polishing compositions (such as "Composition N") pursuant to some embodiments of the present invention comprise an alumina abrasive (alpha-, gamma-, or both), periodic acid ( $\text{H}_5\text{IO}_6$ ), and DI water. Thus, Composition N is comprised of the same components as Composition A. An example of component concentrations for Composition N is set forth below in Table XIV.

#### **Composition N Component Concentration**

**Table XIV: Typical Composition N**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Periodic Acid	0.1 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition N

#### **pH Ranges**

[0091] Composition N advantageously has a pH value of about 1.6.

### Preparation

[0092] Generally, Composition N is prepared by adding water the alumina abrasive (such as CR-30) to a container of DI and subsequently adding the periodic acid ( $H_5IO_6$ ). Composition N is advantageously stirred continuously within the container during at least the period of composition preparation.

### CMP Process

[0093] An example of the mixing ratio, process, pH, removal rates and selectivity associated with Composition N is set forth in Table N.

### Mixing Ratio, Process, pH, Removal Rates, and Selectivity

**Table N**

<b>Mixing Ratio</b>	<b>Process</b>	<b>pH</b>	<b>Removal Rate (A/min) Pt</b>	<b>Removal Rate (A/min) BPSG</b>	<b>Pt: BPSG Selectivity</b>
2 wt % Alpha-Alumina Abrasive	2/200/18/150	1.6	131	180	1:1.5
0.1 mol/l kg Periodic Acid					
DI water					

[0094] In the example set forth in Table N, the CMP process was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Composition N was stirred thoroughly before and during its use.

[0095] When employed according to the process of Table N, Composition N provides a Pt removal rate of 131 A/min and a Boron Phosphorous Silicate Glass (BPSG) removal rate of 180 A/min, demonstrating a Pt:BPSG selectivity of 1:1.5.

### Composition O

[0096] Other platinum polishing compositions (such as "Composition O") pursuant to some

embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, periodic acid ( $\text{H}_5\text{IO}_6$ ), ammonium chloride ( $\text{NH}_4\text{Cl}$ ), and DI water. It is believed that the electrolyte, ammonium chloride, serves as a source of chloride ions that assist in metal etching. One example of component concentrations for Composition O is set forth below in Table XV.

### Composition O Component Concentration

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
Periodic Acid	0.1 mol/1 kg
Ammonium Chloride	0.1 mol/1 kg
DI Water	Remaining weight amount to obtain final desired amount of Composition O

### pH Ranges

[0097] Composition O typically has a pH range from about 1.2 to about 1.8 and a favorable pH value of about 1.6.

### Preparation

[0098] Generally, Composition O is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water, then adding the periodic acid ( $H_5IO_6$ ), and then adding the ammonium chloride. Composition O is advantageously stirred continuously within the container during the composition preparation.

### CMP Process

[0099] An example of the mixing ratio, process, pH, removal rates and selectivity for Composition O is set forth below in Table O.

### Mixing Ratio, Process, pH, Removal Rates and Selectivity

**Table O**

Mixing Ratio	Process	pH	Pt Removal Rate (A/min)	Removal Rate (A/min) BPSG	Pt: BPSG Selectivity



2 wt % Alpha-Alumina Abrasive	2/200/18/150	1.6	443	56	8:1
0.1 mol/kg Periodic Acid					
0.1 mol/kg Ammonium Chloride					
DI Water					

[00100] The CMP process of Table O for Composition O was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Composition O was stirred thoroughly before and during its use.

[00101] Composition O, when employed according to the process of Table O, Composition O provides a Pt removal rate of 443 A/min and a BPSG removal rate of 56 A/min, demonstrating a Pt:BPSG selectivity of 8:1.

[FOR COMPOSITION P, Bob to provide to confirm whether periodic acid concentration is meant to be 0.1 wt %, as moles/kilogram are used elsewhere.]

### Composition P

[00102] Other platinum polishing compositions (such as "Composition P") pursuant to some embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, periodic acid ( $H_5IO_6$ ), ammonium chloride ( $NH_4Cl$ ), and DI water. One example of component concentrations for Composition P is set forth below in Table XVI.

### Composition P Component Concentration

**Table XVI: Typical Composition P**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	6 wt %
Periodic Acid	0.1 wt %
Ammonium Chloride	0.1 wt %
DI Water	Remaining weight amount to

	obtain final desired amount of Composition P
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#### pH Ranges

[00103] Composition P advantageously has a pH range from about 1.5 to about 2.0.

#### Preparation

[00104] Generally, Composition P is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water, then adding the periodic acid ( $H_5IO_6$ ), and then adding the ammonium chloride ( $NH_4Cl$ ). Composition P is favorably continuously stirred within the container during at least the composition preparation.

#### CMP Processes

[00105] Several examples of the processes and removal rates for Composition P are set forth below in Table P.

## Processes and Removal Rates

**Table P**

<b>Process</b>	<b>Pt Removal Rate (A/min)</b>
2/200/18/70	220
4/200/18/70	470
6/200/18/70	750
7/200/18/70	1,020

[00106] The CMP processes set forth in Table P were carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, 4 psi, 6 psi and 7 psi, respectively. All processes further employed a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 70 ml/min. Composition P was stirred thoroughly before and during its use.

[00107] When employed according to the processes set forth in Table P, Composition provides Pt removal rates of 220 A/min, 470 A/min, 750 A/min, and 1,020 A/min, respectively.

## **Composition Q**

[00108] Other platinum polishing compositions (such as "Composition Q") pursuant to some embodiments of the present invention comprise an alumina abrasive (alpha-, gamma-, or both), ammonium chloride (NH<sub>4</sub>Cl), and DI water.

## Composition Q Component Concentration

**Table XVII: Typical Composition Q**

<b>Component</b>	<b>Component Concentration</b>
Alpha-Alumina Abrasive (CR-30)	2 wt %
Ammonium Chloride	0.1 mol/l kg
DI Water	Remaining weight amount to obtain final desired amount of

	Composition Q
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#### pH Ranges

[00109] The present Composition Q advantageously has a general pH range from about 5.0 to about 6.0 and a favorable pH of about 5.4.

#### Preparation

[00110] Generally, Composition Q is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and then adding the ammonium chloride. Composition Q is typically stirred continuously within the container during the composition preparation.

#### CMP Process

[00111] An example of the mixing ratio, process, pH, removal rates and selectivity associated with Composition Q is set forth below in Table Q.

#### Mixing Ratio, Process, pH, Removal Rates and Selectivity

**Table Q**

Mixing Ratio	Process	pH	Pt Removal Rate (A/min)	Removal Rate (A/min) BPSG	Pt: BPSG Selectivity
2 wt % Alpha-Alumina Abrasive  0.1 mol/kg Ammonium Chloride  DI Water	2/0/200/18/150	5.4	1598	145	11:1

[00112] The CMP process set forth in Table Q was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, a back pressure of 0 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Composition Q is advantageously stirred thoroughly before and during its use.

[00113] When employed according to the process of Table Q, Composition Q provides a Pt removal rate of 1,598 A/min and a BPSG removal rate of 145 A/min, demonstrating a

Pt:BPSG selectivity of approximately 11:1.

### **Composition R**

[00114] Other Pt polishing compositions (such as “Composition R”) pursuant to some embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, hydrochloric acid (HCl) and DI water. It is believed that the hydrochloric acid serves as a source of chloride ions that assist in the etching of metal. One example of component concentrations for Composition R is set forth below in Table XVIII.

#### **Composition R Component Concentration**

**Table XVIII: Typical Composition R**

<b>Component</b>	<b>Component Concentration</b>
Alpha-Alumina Abrasive (CR-30)	2 wt %
Hydrochloric Acid	0.1 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition R

### **pH Ranges**

[00115] Composition R typically has a pH range from about 1.0 to about 2.0 and a favorable pH of about 1.2.

### **Preparation**

[00116] Generally, Composition R is prepared by adding the alumina abrasive (CR-30) to a container of DI water and subsequently adding the hydrochloric acid. Composition R is favorably continuously stirred within the container during at least the composition preparation.

### **CMP Process**

[00117] An example of the mixing ratio, process, pH, removal rates and selectivity for Composition R is set forth below in Table R.

Mixing Ratio, Process, pH, Removal Rates and Selectivity

**Table R**

Mixing Ratio	Process	pH	Pt Removal Rate (A/min)	BPSG Removal Rate (A/min)	Pt: BPSG Selectivity
2 wt % Alpha-Alumina Abrasive  0.1 mol/kg Hydrochloric Acid  DI Water	2/0/200/18	1.2	334	26	13:1

CMP Processes:

[00118] The CMP process for Composition R of Table R was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, a back pressure of 0 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Composition R was stirred thoroughly before and during its use.

[00119] When employed according to the process of Table R, Composition R provides a Pt removal rate of 334 A/min and a BPSG removal rate of 26 A/min, demonstrating a Pt:BPSG selectivity of 13:1.

**Composition S**

[00120] Other Pt polishing compositions (such as “Composition S”) pursuant to some embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, hydrochloric acid (HCl), ammonium chloride (NH<sub>4</sub>Cl), and DI water. One example of component concentrations for Composition S is set forth below in Table XIX.

Composition S Component Concentration

**Table XIX Typical Compositions**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %

Hydrochloric acid	0.1 mol/l kg
Ammonium Chloride	0.1 mol/l kg
DI Water	Remaining weight amount to obtain final desired amount of Composition S

#### pH Ranges

[00121] Composition S typically has a pH range from about 1.0 to about 2.0 and, favorably, a pH of about 1.4.

#### Preparation

[00122] Generally, Composition S is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water, then adding the hydrochloric acid, and then adding the ammonium chloride (NH<sub>4</sub>Cl). Composition S is advantageously stirred continuously within the container during at least the composition preparation.

#### CMP Process

[00123] An example of the mixing ratio, process, pH and removal rate for Composition S is set forth in Table S.

#### Mixing Ratio, Process and Removal Rate

**Table S**

<b>Mixing Ratio</b>	<b>Process</b>	<b>pH</b>	<b>Pt Removal Rate (A/min)</b>
2 wt % Alpha-Alumina Abrasive	4/0/200/18/70	1.4	310
0.1 mol/l kg Hydrochloric acid			
0.1 mol/l kg Ammonium Chloride			
DI Water			

[00124] The CMP process set forth in Table S was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 70 ml/min. Composition S was stirred thoroughly before and during its use.

[00125] When employed according to the process set forth in Table S, Composition S provides a Pt removal rate of 310 Å/min.

### **Composition T**

[00126] Other platinum polishing compositions (such as "Composition T") pursuant to some embodiments of the present invention comprise an alumina (alpha-, gamma-, or both) abrasive, hydroxylamine (HDA), and DI water. It is believed that the caustic HDA serves as a mild reducing agent. An example of component concentrations for Composition T is set forth below in Table XX.

### **Composition T Component Concentration**

**Table XX: Typical Composition T**

<b>Component</b>	<b>Component Concentration</b>
Alpha-Alumina Abrasive (CR-30)	2 wt %
Hydroxylamine	0.1 mol/kg
DI Water	Remaining weight amount to obtain final desired amount of Composition T

### **pH Ranges**

[00127] Composition T typically has a pH range from about 8 to about 9 and advantageously a pH of about 8.5.

### **Preparation**

[00128] Generally, Composition T is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and then adding the hydroxylamine. Composition T is



advantageously stirred continuously within the container during the composition preparation.

#### CMP Process

[00129] An example of the mixing ratio, process, pH, removal rates and selectivity for Composition T is set forth below in Table T.

#### Mixing Ratio, Process, pH, Removal Rates and Selectivity

**Table T**

Mixing Ratio	Process	pH	Removal Rate (A/min) Pt	Removal Rate (A/min) BPSG	Pt: BPSG Selectivity
2 wt % Alpha-Alumina Abrasive 0.1 mol/kg Hydroxylamine DI water	4/0/50/51/150	8.5	209	432	1:2

[00130] In the example set forth in Table T, the CMP process was carried out using an IPEC 472 polisher and employing a down force pressure of 4 psi, a back pressure of 0 psi, a table speed of 50 rpm, a carrier speed 51 rpm, and a composition flow rate of 150 ml/min. An IC-1000 k-grooved pad was used as the polishing pad on the primary platen of the polisher, and a Polytex pad was used as buff pad on the secondary platen of the polisher. Composition T was stirred thoroughly before and during its use.

[00131] When employed according to the process of Table T, Composition T provides a Pt removal rate of 209 A/min and a BPSG removal rate of 432 A/min, demonstrating a Pt:BPSG selectivity of 1:2.

#### Composition U

[00132] Other preferred Platinum polishing compositions (such as "Composition U") pursuant to the present invention comprise an alumina abrasive (alpha-, gamma-, or both), hydroxylamine hydrochloride (NH<sub>2</sub>OH·HCl), and DI water. It is believed that the hydroxylamine hydrochloride serves as an oxidizing agent. One example of component

concentrations for Composition U is set forth below in Table XXI.

Composition U Component Concentration

**Table XXI: Typical Composition U**

Component	Component Concentration
Alpha-Alumina Abrasive (CR-30)	2 wt %
NH <sub>2</sub> OH·HCl	0.1 mol/l kg
DI Water	Remaining weight amount to obtain final desired amount of Composition U

pH Ranges

[00133] Composition U typically has a pH range from about 3.5 to about 4.5 and favorably a pH of about 4.0.

Preparation

[00134] Generally, Composition U is prepared by adding the alumina abrasive (such as CR-30) to a container of DI water and then adding the hydroxylamine hydrochloride (NH<sub>2</sub>OH·HCl). Composition U is advantageously stirred continuously within the container during at least the composition preparation.

CMP Process

[00135] An example of the mixing ratio, process, removal rate and selectivity for Composition U is set forth in Table U below.

Mixing Ratio, Process, pH, Removal Rates and Selectivity

Table U

Mixing Ratio	Process	pH	Pt Removal Rate (A/min)	Removal Rate (A/min) BPSG	Pt: BPSG Selectivity
2 wt % Alpha-Alumina Abrasive  0.1 mol/kg NH <sub>2</sub> OH·HCl  DI water	2/0/200/18/150	4.0	393	70	5.6:1

[00136] The CMP process set forth in Table U was carried out using an IPEC 576 polisher with a Thomas West XY pad and employing a down force pressure of 2 psi, a back pressure of 0 psi, a table or platen speed of 200 rpm, a carrier speed of 18 rpm, and a composition flow rate of 150 ml/min. Composition U is advantageously stirred thoroughly before and during its use.

[00137] When employed according to the process of Table U, Composition U provides a Pt removal rate of 393A/min and a BPSG removal rate of 70 A/min, demonstrating a Pt:BPSG selectivity of approximately 5.6:1.

[00138] Those skilled in the art will appreciate that, given the present disclosure, modifications may be made to the invention without departing from the spirit of the inventive concept described herein. Therefore, it is not intended that the scope of the invention be limited to the specific embodiments illustrated and described.

## CLAIMS

1. A composition for chemical mechanical planarization comprising:  
periodic acid and an abrasive in a combined amount sufficient to planarize a material selected from a group consisting of a noble metal, a noble metal alloy, a noble metal oxide, or any combination thereof. **[Bob to provide standards/definitions for “planarize” herein. See bold-faced note in Summary section.]**
2. The composition of claim 1, wherein periodic acid is in an amount in a range of from about 0.05 to about 0.3 moles/kilogram.
3. The composition of claim 1, wherein periodic acid is in an amount in a range of from about 0.075 to about 0.175 moles/kilogram.
4. The composition of claim 1, wherein the abrasive is in an amount in a range of from about 0.2 to about 6 weight percent.
5. The composition of claim 1, wherein the abrasive is in an amount in a range of from about 0.2 to about 4 weight percent.
6. The composition of claim 1, further comprising a pH-adjusting agent in an amount sufficient for a pH level of the composition to be in a range of from about pH 5 to about pH 9. **[Bob to clarify pH range, especially considering IrO<sub>2</sub> examples.]**
7. The composition of claim 6, wherein the pH-adjusting agent is selected from a group consisting of tetramethylammonium and ammonium hydroxide. **[Bob to name other pH-adjusting agents, such as particular quaternary amine salts, choline, etc.]**
8. The composition of claim 1, further comprising a pH-adjusting agent in an amount sufficient for a pH-level of the composition to be in a range from about pH 1 to about pH 4.

9. The composition of claim 1, further comprising a pH-adjusting agent in an amount sufficient for a pH-level of the composition to be in a range from about pH 2 to about pH 3.

10. The composition of claim 8, wherein the pH-adjusting agent comprises ammonium hydroxide. **[Bob: Any other pH-adjusting agents for this particular claim?]**

11. The composition of claim 1, further comprising a suspension agent.

12. The composition of claim 11, wherein the suspension agent is selected from a group consisting of an organic acid, a surfactant, another abrasive, and ethyl carbonate.

13. The composition of any of claims 1 through 12, wherein the abrasive comprises alumina.

14. The composition of any of claims 1 through 12, wherein the layer comprises a material selected from a group consisting of Ir, IrO<sub>2</sub>, Pt, and any combination thereof.

15. The composition of claim 14, wherein the abrasive comprises alumina.

16. A method of planarizing a substrate layer comprising a noble metal, a noble metal alloy, a noble metal oxide, or any combination thereof, comprising:

providing a composition, the composition comprising periodic acid and an abrasive in a combined amount sufficient to planarize the layer; and

planarizing the layer with the composition. **[Bob to provide standards/definitions for “planarize” and “planarizing” herein. See bold-faced note in Summary section.]**

17. The method of claim 16, wherein periodic acid is in an amount in a range of from about 0.05 to about 0.3 moles/kilogram.

18. The method of claim 16, wherein periodic acid is in an amount in a range of from about 0.075 to about 0.175 moles/kilogram.

19. The method of claim 16, wherein the abrasive is in an amount in a range of from about 0.2 to about 6 weight percent.

20. The method of claim 16, wherein the abrasive is in an amount in a range of from about 0.2 to about 4 weight percent.

21. The method of claim 16, wherein said providing comprises providing a composition that further comprises a pH-adjusting agent in an amount sufficient for a pH level of the composition to be in a range of from about pH 5 to about pH 9. **[Bob to clarify pH range, especially considering  $\text{IrO}_2$  examples.]**

22. The method of claim 21, wherein the pH-adjusting agent is selected from a group consisting of tetramethylammonium and ammonium hydroxide. **[Bob to name other pH-adjusting agents, such as particular quaternary amine salts, choline, etc.]**

23. The method of claim 16, wherein said providing comprises providing a composition that further comprises a pH-adjusting agent in an amount sufficient for a pH-level of the composition to be in a range from about pH 1 to about pH 4.

24. The method of claim 16, wherein said providing comprises providing a composition that further comprises a pH-adjusting agent in an amount sufficient for a pH-level of the composition to be in a range from about pH 2 to about pH 3.

25. The method of claim 23, wherein the pH-adjusting agent comprises ammonium hydroxide. **[Bob: Any other pH-adjusting agents for this particular claim?]**

26. The method of claim 16, wherein said providing comprises providing a composition that further comprises a suspension agent.

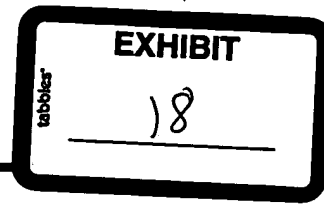
27. The method of claim 26, wherein the suspension agent is selected from a group consisting of an organic acid, a surfactant, another abrasive, and ethyl carbonate.

28. The method of any of claims 16 through 27, wherein the abrasive comprises alumina.

29. The method of any of claims 16 through 27, wherein the layer comprises a material selected from a group consisting of Ir, IrO<sub>2</sub>, Pt, and any combination thereof.

30. The method of claim 29, wherein the abrasive comprises alumina.

**de Runtz, Alison**



**From:** de Runtz, Alison  
**Sent:** Friday, November 16, 2001 10:49 AM  
**To:** 'Bob\_Small/EKC/FCC/First\_Mississippi\_Corporation@chemfirst.com'  
**Subject:** FW: Status Report for ChemFirst, Inc

-----Original Message-----

**From:** de Runtz, Alison  
**Sent:** Friday, November 16, 2001 10:33 AM  
**To:** 'Bob\_Small/EKC/FCC/First\_Mississippi\_Corporation@chemfirst.com'  
**Cc:** 'rluke@chemfirst.com'  
**Subject:** Status Report for ChemFirst, Inc



HJD6011\_.doc

Dear Bob:

Attached please find an updated status report for ChemFirst's US patent portfolio. Today, we are sending you a second draft of the 20603-pa application for your review. Please see the bold-face text regarding information needed for disclosure in this 20603-pa application, which we propose submitting when we file that application.

I look forward to hearing from you shortly concerning the 20603-pa draft application.

Best regards, Alison.



de Runtz, Alison



**From:** de Runtz, Alison  
**Sent:** Monday, November 26, 2001 2:34 PM  
**To:** 'BSmall@ekctech.com'  
**Subject:** RE: Hello

Thank you much, Bob. We'll incorporate the material you will be sending into a third draft for your review, and when we've arrived at a finalized application, we'll send you formal documents for you and Zhefei to sign. We look forward to receiving your new material shortly.

Best regards, Alison.

-----Original Message-----

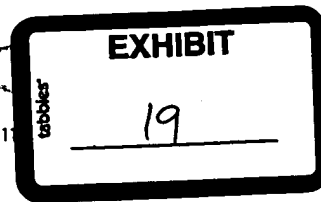
**From:** BSmall@ekctech.com [mailto:BSmall@ekctech.com]  
**Sent:** Monday, November 26, 2001 2:18 PM  
**To:** de Runtz, Alison  
**Subject:** RE: Hello

Here is Jessie Chen's information.

Bob

3 Embarcadero Center  
28th Floor

San Francisco  
California 94111



San Jose, CA  
Austin, TX  
Newport Beach, CA

skjervemorrill  
macpherson LLP

December 20, 2001

**FILE COPY**

VIA Federal Express

Robert J. Small, Ph. D.  
CMP Technical Director  
EKC Technology, Inc.  
2520 Barrington Court  
Hayward, CA 94545-1163

Re: U.S. Patent Application entitled: "Compositions For Chemical-Mechanical  
Planarization Of Noble-Metal-Featured Substrates, Associated Methods,  
And Substrates Produced By Such Methods"

Inventors: Robert J. Small and Zhefei J. Chen

Your Reference: 20603-pa

Our Reference: M-12178 US

Dear Bob:

Enclosed please find a further revised draft of the above-referenced patent application, which incorporates the new material you recently provided. Please review the draft application carefully for completeness and accuracy, and particularly review the bracketed and bold-faced text as to items requiring your further input.

Upon your review of the draft application, please telephone me so we can discuss finalizing the application with your further information. I look forward to hearing from you at your earliest convenience, so that we can get this application on file as early as possible.

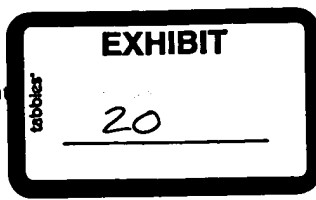
Very truly yours,

K. Alison de Runtz

ADR/rp  
Enclosure

830958 v1

3 Embarcadero Center  
28th Floor



San Jose, CA  
Austin, TX  
Newport Beach, CA

skjerven morrill  
macpherson LLP

January 18, 2002

**FILE COPY**

VIA Federal Express

Robert J. Small, Ph.D.  
CMP Technical Director  
EKC Technology, Inc.  
2520 Barrington Court  
Hayward, California 94545-1163

Re: U.S. Patent Application entitled "Compositions For Chemical-Mechanical Planarization Of Noble-Metal-Featured Substrates, Associated Methods, And Substrates Produced By Such Methods"

Inventors: Robert J. Small and Zhefei J. Chen

Your Reference: Not yet assigned

Our Reference: M-12178 US

Dear Bob:

Enclosed is the ready-to-file patent application for your above-mentioned invention. Please review this application for accuracy and completeness. Please review the application carefully to make sure it fulfills the following criteria:

1. accurately reflects your invention;
2. contains a written description of your invention and the manner and process of making and using it;
3. describes your invention in sufficient detail to enable one skilled in the art to make and use your invention; and
4. discloses the best mode contemplated by you of carrying out your invention.

If the application meets these criteria and is acceptable for filing in the United States Patent and Trademark Office without any changes, please sign the following enclosed documents.

DOCUMENTS TO BE SIGNED:

- ⇒ Declaration for Patent Application – should be signed and dated by you and Zhefei Chen as the inventors.
- ⇒ Assignment – should be signed and dated by you and Zhefei Chen as the inventors before a notary public.

Robert J. Small, Ph.D.  
EKC Technology, Inc.  
M-12178 US  
January 18, 2002  
Page 2

When an inventor signs the declaration, the inventor represents that he or she has reviewed and understands the specification including the claims. Failure to read and understand the specification, including the claims, before signing the declaration may jeopardize the application as well as any patent or patents which issue from the application.

**DOCUMENTS TO BE RETURNED TO US FOR FILING IN U.S. PATENT AND TRADEMARK OFFICE ("USPTO"):**

The Declaration, Assignment, application, and drawings should be returned to us. We will then file these documents in the U.S. Patent and Trademark Office.

**IF CHANGES ARE REQUIRED:**

If changes or additions are required in the application, please call me immediately so that we can decide how these changes or additions can best be made.

**APPLICANTS' DUTY OF CANDOR TO USPTO:**

Please keep in mind that a patent applicant is required by law to bring to the attention of the U.S. Patent and Trademark Office all prior art and prior acts (articles, publications such as books, any devices used, sold or offered for sale) of which the applicant is aware and which are relevant to the applicants' invention. If you are aware of any such prior art or acts, now or in the future, please let us know as soon as possible. If you or Zhefei are in doubt as to whether something is prior art or relevant to the above invention, please call us and we can help you decide.

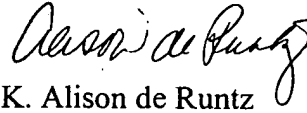
**FILING THE APPLICATION OUTSIDE THE UNITED STATES:**

Please let us know if you wish to file this application outside the United States. If you believe this invention is sufficiently important to file abroad, you should do so within one year following the United States filing date. Because of the time involved in obtaining translations and preparing the filing papers, you should make a decision as to foreign filing no later than nine months following the United States parent filing date. Note that in most (but not all) "non-convention" countries, you cannot gain the benefit of the United States filing date. If you are interested in filing the application in a non-convention country, generally you must do so prior to any public use, publication, or other public disclosure of the invention anywhere in the world. If you do not know whether one or more countries in which you intend to file the application outside the United States are convention or non-convention countries, please contact us immediately. Exceptions to this are India, Taiwan, and Thailand which, while not Paris Convention countries, will recognize the U.S. filing date as the filing date in India, Taiwan, or Thailand if the case is filed within one year of the U.S. filing date for this patent application.

Robert J. Small, Ph.D.  
EKC Technology, Inc.  
M-12178 US  
January 18, 2002  
Page 3

If you have any questions on this matter, please contact me immediately.

Very truly yours,



K. Alison de Runtz

ADR/rp  
Enclosure

838304 v1

cc: R. B. Luke (w/enclosures)